TOTAL SOLIDS & MOISTURE

MASON

FAT, TOTAL SOLIDS AND MOISTURE.

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FAT, TOTAL SOLIDS AND MOISTURE

DETERMINATIONS BY THE "TECHNICO" TEST UNIT.

By

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Contents.

Introduction.

MUCH has been written and considerable work undertaken concerning the determination of moisture and total solids in materials of all kinds, especially foods.

The literature bears witness to the controversial nature of this subject but opinion seems now to incline to some form of vacuum oven technique, which, however, is not by any means standardised.

The main disadvantages of any method other than a " vacuum " oven method is the time consumed in arriving at concordant results, many hours usually being required.

In many proposed " vacuum " methods, of course, this criticism also applies and it seems that prolonged heating periods would tend to promote degradation in some form and hence from this point of view alone would be undesirable; but there is less likelihood of degradation when the " vacuum " oven method is employed.

A brief review of the more recent work carried out on the subject might not be entirely out of place here and would illustrate the trend of opinion.

In 1925, G. A. Shuey[1] published work in which he states that employing a vacuum oven method in which the substance is heated at 98-100°C. at a pressure not exceeding 25 mms. of mercury for a period of five hours he obtained slightly higher results than the three test methods he employed in which the substance was heated in an air oven at temperatures of 125°C., 130°C. and 135°C. for 1 hour.

(1) *Cereal Chemistry*, 1925, 2, 318.

Work was carried out by J. H. Lanning[2] in 1935 in which he used a vacuum oven method as a reference when he was examining the effect, amongst other things, of temperature on moisture determinations. The range of temperatures employed was 110-140°C., and between these limits large variations in results were experienced.

Again, in the same year, C. F. Davies[3] came to the conclusion that the introduction of forced air-draught contributed little if anything to the reliability of the official air-oven procedure and as ventilation conditions were found to have very little effect on the results obtained he argued that this strengthened the position of the air-oven method as one of reference.

A. E. Treloar and B. Sullivan[4] in the same year studied statistically the results obtained by collaborators working for the Methods Committee for the year 1934-1935 using three different methods: these were (a) the electric air-oven method working at 130°C. for 1 hour using 2 grams of sample, (b) vacuum oven method working at 98-100°C. for five hours at 25 mms. pressure employing 2 grams sample, (c) the electric air-oven method working at 135°C. for 2 hours employing 2 grams of sample.

From the results of this study they came to the conclusion that greater consistency was to be obtained by means of the vacuum oven method than by means of either of the others, which they suggest may be due to the superiority of the method, although they cite other possibilities which could easily account for such consistency of result.

In the same volume of that *Journal*,[4] D. A. Coleman and S. R. Snider presented a paper dealing with the determination of moisture in barley malt. In their work, they employed ten different

(2) *ibid.*, 1935, **12**, 69.
(3) *ibid.*, 1935, **12**, 512.
(4) *ibid.*, 1935, **12**, 520.

methods and studied the results obtained in the light of the varying experimental conditions attendant upon each method. They employed as their standard reference method that given in the "Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists," (3rd Edition, p. 278, sec. III, paras. 5 and 6), with the exception that they substituted phosphorus pentoxide for sulphuric acid. This method was adopted as a standard partly because there could be no possibility of caramelisation and partly because only "free" and not "bound" water would be likely to be removed.

The test methods examined consisted of the air-oven method operating at 130°C., the Bidwell-Sterling method, the Carter-Simon method, the standard vacuum oven method, the standard method of Malt Analysis, the vacuum oven method working at 25 mms. of mercury for 18 hours at a temperature of 70°C., the water-oven method working at the temperature of boiling water for five hours, the Brown-Duvel method, and the Cambridge dielectrically operated moisture meter method.

As an outcome of this lengthy paper, the authors state that, amongst other findings, the method employing a vacuum oven operated at 25 mms. pressure at a temperature of 100°C. for a period of 5 hours gives results which are 0.25% higher than the method of the vacuum desiccator which they chose as their standard reference.

In a review of "Progress of Cereal Chemistry," Clinton Brooke[5] makes reference to the air-oven method and alludes to the defence of the method put forward by Köster in 1936. In this defence, Köster shows that the results obtained by the method employing 130°C. as the heating temperature for 1 hour are less affected by temperature

(5) *ibid.*, 1936, **13**, 367.

fluctuations and variations in the relative humidity, and gives results more nearly approaching the theoretical value than those obtained by the method of heating for 105°C. for 3-8 hours.

In the same article, Clinton Brooke refers to the findings of Tucker and Burke with reference to the distillation method using tetrachlorethane. According to these workers the values obtained are usually higher than those obtained by drying at 98.5°C. and are unreliable in the presence of large quantities of reducing sugars.

J. E. Anderson in a paper on "Some Facts Concerning Vacuum-Oven Moisture Determination"[6] discusses the various factors involved, including such important points as the gain or loss of moisture during weighing, the length of time required for drying, the temperature to which the samples are heated in the vacuum oven, the effect of reduced pressure in the oven chamber on the temperature of the sample and the use of steam-heated shelves in the vacuum oven.

In his conclusions he states that errors are considerably reduced by fitting the dishes with loosely fitting lids and admitting only dry air to the oven when "breaking" the vacuum, this procedure reducing the gain or loss incurred during weighing. It was also found that 16-24 hours drying were needed in the ordinary type of air-jacketed vacuum oven to yield results comparable with those obtained in a vacuum oven employing steam-heated shelves with a heating period of from four to six hours.

He also makes the important point that the final results obtained even under vacuum oven conditions depend upon the ratio of the vapour pressure of the atmosphere at the time of the determination to the vapour pressure of the moisture remaining in the material.

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(6) *ibid.*, 1936, 13, 437.

Anderson, in the same conclusion, also draws attention to the fact that a load of dishes heats up to oven temperature in the steam-heated shelf oven in a very small fraction of the time that it requires for the same load to heat up in the air-jacketed vacuum oven.

The question of vapour pressure and humidity is also examined very thoroughly by Ludwig Papp in a paper entitled "Influence of Humidity on Moisture Determination," in which he illustrates the magnitude of the effects of vapour pressure on moisture determinations and gives a method for the correction of results for varying vapour pressures.

He states in this paper that moisture values are influenced by temperature, vacuum, thickness of the layer of the material to be dried, size of the particles of the substance to be dried and the position of the dish in the drying oven.

He illustrates the type of variation in result which is likely to be experienced in moisture determination with the following figures—50% of relative humidity at 20°C. corresponds to a relative vapour pressure of 0.97 at 105°C. and 1.38 at 95°C., the corresponding moisture contents being 13% and 12.73% respectively.

The following results taken from the same paper serve to emphasise the effect of the influence of varying humidities on moisture determinations conducted at three temperatures:—

Temperature of Determination	*Relative Humidity at 20°C.*	*Determined Moisture Content*
95°C.	20%	13.15%
	40%	12.88%
	60%	12.60%
	80%	12.33%
	100%	12.05%

Temperature of Determination	*Relative Humidity at 20°C.*	*Determined Moisture Content*
105°C.	20%	13.45%
	40%	13.08%
	60%	12.90%
	80%	12.72%
	100%	12.55%
115°C.	20%	13.38%
	40%	13.25%
	60%	13.15%
	80%	13.02%
	100%	12.90%

He summarises his work by stating that with cereal products the limits of error due to variable humidity can range from ±0.45% at 95°C. down to ±0.2% at 115°C.

In reviewing this brief historical survey of the literature it appears that considerable diversity of opinion exists as to which should be adopted as a standard method for the determination of moisture, but there would appear to be some weight of opinion in favour of the vacuum oven method even with all the varied conditions advocated by various workers.

In view of this diversity of opinion as to what should constitute a standard method for the determination of moistures and total solids, it is obviously unwise to postulate a reference standard in work of this kind. The only criterion the worker has available is his old method previously used, and to compare the results obtained by both methods, old as well as new, is the only way he can ascertain the reasonableness of his results obtained by the new method.

Many chemists may raise objection to these statements, saying that the absence of direct comparison of results with those obtained by a standard method invalidates the new method.

It is felt that any such criticism has been answered by the demonstration in the foregoing

words of the utter confusion which exists at the moment as to what constitutes a standard and therefore to publish such comparative figures would only tend to arouse controversy amongst individual workers each justly considering the method he is using at the moment as much entitled to be judged a standard method as the one singled out by the author of this handbook.

It might be said in passing that the method adopted in the author's laboratories has been, in the past, the electrically-heated, thermostatically-controlled air oven, with special side ventilation, operating at 105°C. for 1 hour: in practically every case examined, the results for moistures were higher by the vacuum oven method than by the electrically heated oven method.

In order to demonstrate the excellent reproducibility obtainable by the vacuum oven method, some actual results obtained on one or two foodstuffs are here included. These results not only serve to show that it is possible to obtain excellent duplicates, but they will be all the more enlightening when it is known that they were obtained by junior assistants in the author's laboratories who were speedily trained to undertake the work: the results were obtained by the methods given in this handbook.

Cornflour (Moisture Content)					
%	%	%	%	%	%
14.00	14.00	14.15	12.75	13.30	12.55
14.00	14.00	14.15	12.70	13.30	12.55

Soups and Stocks (Total Solids Content)					
%	%	%	%	%	%
6.19	5.12	6.40	3.46	1.63	1.62
6.19	5.13	6.36	3.47	1.65	1.63

Biscuits (Moisture Content)			
%	%	%	%
5.00	3.90	3.32	2.71
4.90	3.90	3.23	2.76

It is significant that the Association of Official Agricultural Chemists have included as an official method in their " Tentative Methods " the determination by means of the vacuum oven of moistures and total solids in a number of cereal products such as flour, bread, macaroni, etc., and the same method has been similarly adopted by the American Association of Cereal Chemists.

If for no other reasons than these, there would appear considerable justification for attempting to adapt the vacuum oven method in one form or another to the determination of moistures and total solids on account of the numerous advantages attaching to it over most other methods. These advantages can briefly be enumerated as follows:—

(1) Economy of time for each determination.
(2) Less risk of aerobic oxidation due to prolonged heating in air.
(3) Lower temperatures may be employed, thereby reducing the dangers of possible thermal decomposition.
(4) The effects of varying conditions of humidity are reduced to a minimum.
(5) The liberated moisture is removed speedily from the heating chamber.

These undoubted advantages were recognised in the writing of this little handbook and the " Technico Test Unit " was used in part as the medium by which the methods which follow in later pages were built up together with the experience gained by the author on an apparatus of similar design.

In the experience of the author, based upon many years' work with vacuum-type electrically-heated ovens, elevated temperatures and long periods of heating are not necessary and it has been found that in the majority of cases a temperature of 100°C. approximately for a period of about one hour is admirably suited to the determination of moisture and total solids in most substances.

No attempt has been made to prepare an exhaustive account of the application of the "Technico" Test Unit to all food products.

What has been attempted, however, is to set down a number of examples of products of a diverse nature in order to indicate the immense possibilities of the apparatus when applied to the determination of fats and moisture, or, alternatively, total solids. Much work remains to be done on the extension of the use of the apparatus to many products, not necessarily food products, and this can only be achieved by careful investigation of the conditions as applied to each individual item attempted.

Although the methods given in this book are directly concerned with foodstuffs, the comparatively low temperature and short time of heating should recommend the adoption of vacuum oven technique to workers in other fields, such as the paint, textile and cement industries, to quote only a few possibilities.

It will be noticed that much of the detailed instruction is repeated under each individual heading, especial stress being laid upon such practical points as the flowing of the cooling fluid in the desiccator, the quantities of extractants to be employed for the determination of fat, etc. This has been done for a special reason.

So much time is lost, and irritation occasioned by the necessity of constant reference to instructions given in previous pages of a methods book, that it is felt that each particular method should be, in itself, as complete as possible to prevent this constant cross reference: some cross reference is, of course, inevitable, but efforts have been made to reduce this to the barest minimum.

Again, some explanation is necessary for the division made between the section dealing with the method for the determination of total solids and that dealing with moisture determination. In the determination of total solids it has been found by

experience that nickel dishes are preferable to aluminium dishes, on account of the corrosion of the aluminium during the estimation of total solids where acid products are being examined. The technique involved in each case is also different.

The methods dealing with milk products are taken in the main from "The Technical Control of Dairy Products," by Mojonnier and Troy, Second Edition, 1925, and reference is also made to the work carried out by Lampitt, Hughes and Bogod in their paper on the "Routine Examination of Dairy Products."(7)

The methods for the determination of total solids in fruit products are based on the work of Hughes and Maunsell, an account of which appears in the *Analyst* on "The Analysis of Fruit and Fruit Products."(8)

In many instances the methods of sampling are based upon the recommendations laid down in the "Official and Tentative Methods of Analysis of the A.O.A.C.", 3rd and 4th editions, and in the Journal of the Association of Official Agricultural Chemists.

Grateful acknowledgment and thanks are here accorded to The Society of Public Analysts and Other Analytical Chemists, The Association of Official Agricultural Chemists, The American Association of Cereal Chemists and the American Chemical Society for the permission so readily and willingly granted to quote from their journals "*The Analyst*," "*Official and Tentative Methods of Analysis of the A.O.A.C.*" (3rd and 4th editions), "*Journal of the Association of Official Agricultural Chemists*," "*Cereal Chemistry*," "*Cereal Laboratory Methods*," and the "*Journal of Industrial and Engineering Chemistry*."

Much of the work set down here has also been the result of intensive investigation by the author

(7) *Analyst*, 1924, **49**, 414.
(8) *ibid.*, 1934, **59**, 231.

and his assistants in the Laboratories of Messrs. W. J. Barton Ltd., to whom the author is indebted for permission to publish.

Finally, indebtedness is expressed to Mr. H. J. Hornby, of Messrs. A. Gallenkamp & Co., Ltd., for his invaluable help in the matter of publication and the provision of suitable blocks for the illustrations required, and to Mr. E. G. Purser, B.Sc., A.I.C., for his helpful criticism of the text and his painstaking reading of the proofs.

Bickley, Kent, 1939.

R. D. M.

CHAPTER I.

THE "TECHNICO" FATS AND TOTAL SOLIDS TEST UNIT.

General Description.

The "Technico" Test Unit has been designed to enable rapid determinations of fat and moisture in many types of foodstuffs to be carried out with accuracy, employing in the case of fat determinations the modifications of the Röse-Gottlieb method.

Briefly, the main principle underlying the unit is the acceleration of the time-consuming operations normally entailed in fat and total solids determinations, such as evaporating and drying.

The method of extraction of the fat in any substance under investigation is accelerated by the use of the centrifuge, while the evaporation and drying operations are speeded up by the use of hot-plate vacuum ovens: the cooling processes are likewise expedited by the use of desiccators cooled by a special emulsion circulated through the supporting base plate.

The Unit is so designed that the determinations of both fat and total solids may be carried out simultaneously by one operator without his undue movement from the centre of operations, a factor which makes for speed and renders the unit admirably adapted to routine work.

The unit throughout is of British design and manufacture and will give trouble-free service with maximum efficiency.

The external finish of white enamel and chromium plating presents a clean and pleasing appearance.

The unit has been so constructed that all its component parts have been standardised and can, in consequence, be supplied from stock when required.

The illustration shows the Unit complete and a full description of the essential features is given below.

1. Section upon which all fat determinations are carried out.
2. Section upon which all total solids determinations are carried out.

3 & 6. Cooled desiccators with exteriors white stove-enamelled, fitted with hinged doors having precision-ground surfaces.

4 & 5. " Fats " and total solids vacuum ovens both electrically heated and thermostatically controlled. Each oven is cast from virgin pig iron, sherardized and covered externally with white stove enamel, while the metal parts are all heavily chromium plated.

These ovens are heated by hot-plates of mild steel, machined flat and cadmium plated. The heating is accomplished by elements situated inside the plate and so spaced as to give uniform heating over the entire surface. The thermostat is of the standard pattern, very robust, and giving accurate and fine control working in conjunction with a vacuum switch, thus eliminating noise during operation.

The " fats " and " solids " ovens are thermostatically controlled at 135°C. and 105°C. respectively. Each oven is also furnished with a tray fitted underneath the hot plate and containing a desiccant in order to reduce the vapour pressure of the exhausted atmosphere to a minimum at the time of a determination. Proximity to absolute results is thereby more nearly attained (Ludwig Papp).[9]

The areas of the hot plates are 112 sq. ins. and the working space in the ovens is 330 cubic inches.

Each oven is capable of being exhausted either separately or together by the electrically operated pump to a ' vacuum ' of about 28-30 ins. The degree of ' vacuum ' is indicated by a high grade gauge fitted to each oven and controlled each by a grease-filled vacuum tap.

7 & 8. Total solids and " fats " hot-plate evaporators : these are constructed in exactly the same way as the hot plates in the vacuum ovens, but in this instance the plates are dull nickel plated. They measure 15 ins. by 7½ ins. and again are carefully machined flat.

The " fats " hot-plate is thermostatically controlled at 135°C. and the total solids hot-plate at 180°C.

9 & 10. Gauges for recording vacua in the two heating ovens.

11. Burettes fitted each to its appropriate reservoir. The reservoirs are of resistance neutral glass and are clamped together in the metal framework. All the burettes are simply and rapidly filled by tipping the frame forward by means of the handle on the right, the frame pivoting on two plain bearings in the supporting arms. The levels of the various reagents are automatically adjusted to the zero marks by constant level return arms communicating from the sides of the burettes and passing directly into the reservoirs.

Each reservoir has a capacity of three litres and each is fitted with a ground-in glass stopper, and carries on its surface a sand-blasted label to indicate the contents.

(9) *Zeitschrift* G.M.B. No. 6, 1934.

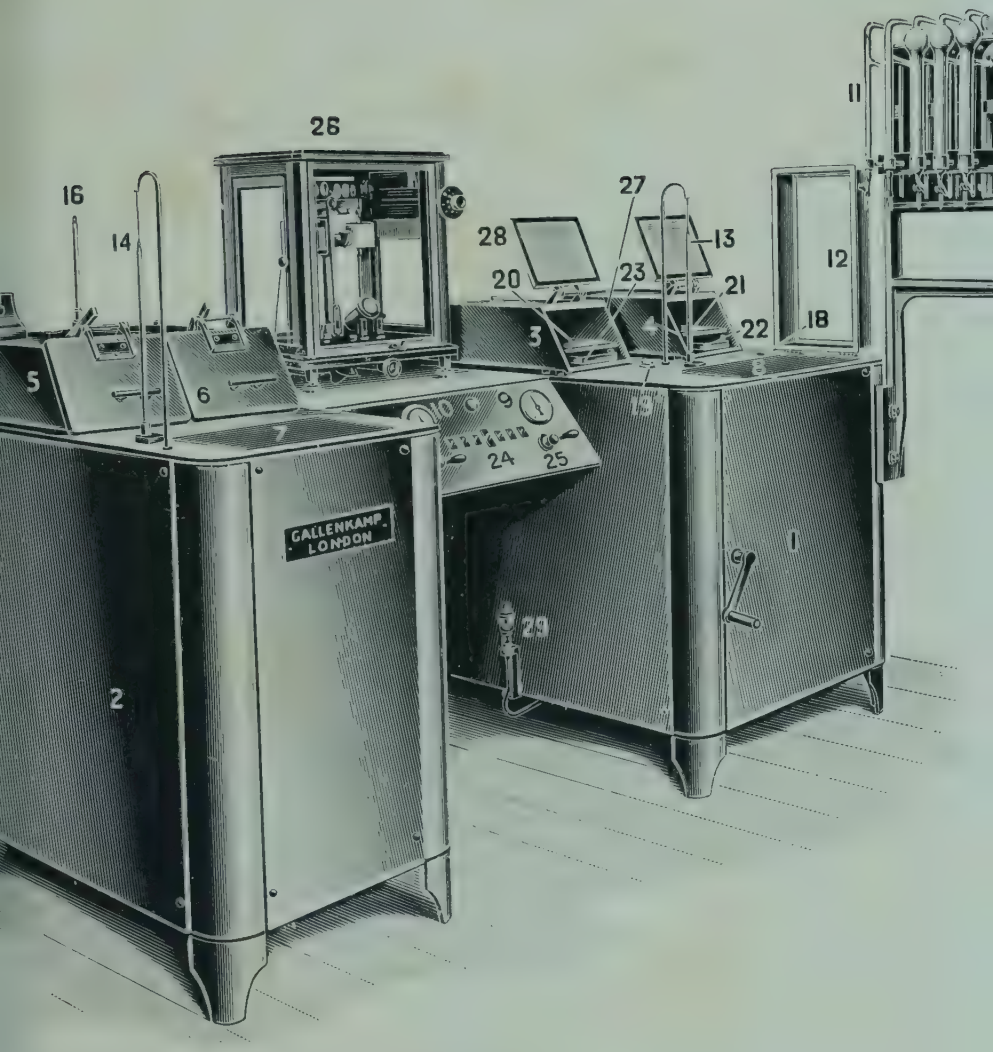

FIG. 1.

The "Technico" Test Unit for the Determination of Fat, Total Solids and Moisture.

The burettes containing distilled water and ammonium hydroxide are graduated in 0.5 ml. divisions; those containing ether and petroleum ether are graduated in 5.0 ml. divisions. The containers are arranged from left to right in the order in which they are used, viz.: water, ammonium hydroxide, alcohol, ethyl ether and petroleum ether.

12. Metal-framed glass-topped hood hinged to the platform of the " fats " section and arranged to cover the " fats " hot-plate. By the side of the hot-plate is a vent connected to the exhaust pump: thus when the hood is lowered over the hot-plate all vapours generated by evaporation of solvents during fat determination are drawn speedily away through this vent.

13 & 14. Thermometers with mercury wells provided for checking the temperatures of both " fats " and total solids external hot-plates.

15. Thermometer dipping into a mercury well situated inside the oven for total solids determinations: this provides check temperature readings against the thermostat. A similar thermometer is provided for the " fats " oven. The level of the mercury in these cups should be carefully checked about once a month.

16. Thermometer dipping into a mercury well situated inside the desiccator for total solids determinations.

17. Thermostatic control " tell-tale " light and adjustment.

18. Aperture through which vapours from the volatile solvents are sucked to waste from the " fats " hot-plate.

19. Flush-fitting flap on the " fats " section allows access to the centrifuge which is provided with two buckets of heavily tinned copper, each capable of carrying four fat extraction flasks. (The hand-operated type of centrifuge is brought to rest by means of the handle.)

The whole is illuminated for inspecting the extraction flasks to facilitate insertion and withdrawal of the flasks into and from the buckets.

Electrically-driven centrifuges with a " start " and " running " switch can be provided operating at a constant speed of 500 r.p.m.

20. Cooling plate made of 20 gauge brass, nickel plated. The special emulsion or cooling fluid is fed in at one end and circulated through the plate by means of a pressure pump on the motor unit.

21. Hot-plate.

22. Tray for desiccant.

23. A removable tray 13 ins. by 4 ins. situated under the cooling plate and containing a suitable desiccant such as silica gel.

24. Switches and " tell-tale " signal lights controlling hot-plates and the heating elements of the vacuum ovens: the signals show red during any period when the heating elements are in operation, and are extinguished when the thermostatic control cuts out. This system eliminates the danger of the ovens being used until they have attained the right temperatures.

25. Vacuum taps, grease filled, with conical ground plungers.

26. The balance.

27. Precision ground oven door surface.

28. Special vacuum tight machined-flange hinged doors.

29. Oil-drip feed to pump.

The Power Unit.

The motor unit, which is a $\frac{1}{3}$ h.p., is compact and efficient and supplies power for the vacuum pump and the cooling fluid circulating pump, both of which are connected with V-belt drives.

A good vacuum is established speedily by means of the "Technico" vacuum pump. This runs in a generously proportioned oil-bath, and it can be left running under load indefinitely without attention and without danger of over-heating: it is fitted with a non-return valve to prevent "sucking-back" of oil into the vacuum lines. Wakefield's special vacuum oil is used, a small quantity of which is supplied for "topping-up" purposes.

The tank mounted above the motor supplies the cooling fluid to the desiccators for cooling purposes via the pump; the cooling fluid is prepared by treating water to the extent of 10% with "soluble" oil, which is added to prevent freezing and to help in the lubrication of the pump.

A sight drip-feed oiler supplies lubricant to the gland on the pulley-end and should not be allowed to run dry as this would cause overloading of the motor which would eventually burn out.

The electrical circuits are controlled from a panel situated underneath the balance table. Indicating lamps are used on the main switch and one is used for each of the thermostatically controlled hot-plates: when alight they indicate the hot plates are heating up and when extinguished that the hot-plates are cooling.

The only connections necessary are the three-core T.R.S. Cable to the main supply and those connecting the exhaust pump to a flue which carries the fumes outside the room in which the determinations are made.

Each section has four panels which are detachable for inspection of the mechanism underneath.

The makers have recognised that many chemists will not require the complete Unit as described above and provisions have been made for the marketing of two separate sections:—

(*a*) Section No. 1, comprising the total solids

vacuum oven, desiccator and hot-plate with the necessary accessories, complete with power unit.

(*b*) Section No. 2, comprising the fats vacuum oven, desiccator and hot-plate, complete with power unit and necessary accessories.

By means of this arrangement it is possible for a laboratory engaged upon work entailing many routine tests involving either total solids, moistures, or fat determinations, to avail itself of the section it requires without incurring the cost of the complete Test Unit.

The Balance.

For the type of work undertaken on the "Technico" Test Unit, rapid as well as accurate weighings are essential and for this reason the "quick-stop" direct reading or the "chainomatic" class of balance is most suitable.

Careful provision has been made for the mounting of the balance between the two units. An adjustable "Technolite" plate, supported by two cross members on special cork and rubber pads, is provided in order to insulate the balance from vibration or accidental shock.

General Care of the Tester.

1. Keep all japanned parts clean with soap and water.

2. Keep the water tank of the cooling unit filled completely—if this is not done cooling will be inadequate. See that the correct amount and grade of "soluble" oil is added.

3. Keep the vacuum pump chamber filled with the correct grade of oil.

4. The electric motor requires no less and no more attention than any ordinary motor: clean and lubricate every year according to the instructions.

5. "Top up" the thermometer wells in the vacuum ovens with mercury at least once a month: make a periodic inspection of the others.

6. Do not allow mercury to come into contact with the hot-plates under any circumstances: if any is accidentally spilled remove at once.

7. Do not allow mercury to come into contact with the aluminium dishes.

8. Keep the ground faces of the doors and vacuum ovens scrupulously clean. Failure to maintain these faces in proper condition is the most frequent cause of poor vacua.

A very slight smear of vaseline sometimes helps to obtain a good seal, but generally this should not be necessary.

9. Check thermometer seals of the vacuum ovens periodically for leaks.

Instructions for Starting up the Tester.

To commence work with the tester the following operations must be carried out:—

1. Switch on main power switch and sub-switches for the heating elements of the hot-plates and vacuum ovens.

2. Wait until the red pilot signal is extinguished, a period of about 30 minutes, when the ovens will have attained their correct respective temperatures of 105°C. and 135°C.

3. Read the hot-plate temperatures by moving the mercury wells on to the hot surfaces and continue to note the readings until a constant temperature is attained. If the temperatures are not correct (180°C. or 100°C. for solids plate and 135°C. for fat plate) adjust the thermostatic control until the correct conditions are established.

4. Make sure the reagent burettes are filled to their respective zero marks.

5. When the ovens have attained equilibrium conditions, place the load of clean, dry and empty

Fig. 2

Moisture or "Solids" Dish showing lid used for moisture determination.

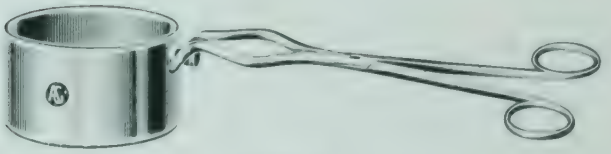

Fig. 3

Dish for Fat Determination showing special side fitting for ease of handling with tongs.

Fig. 4

Method of Use of the Dish Depressor.

fats dishes in the " fats " vacuum oven and the load of " solids " dishes and their respective lids in the " solids " vacuum oven in preparation for the initial weighings.

6. Close the vacuum ovens' doors firmly and switch on the motor so that during this preparation period the ovens are under full " vacuum."

A. General Outline of the Test for the Determination of Total Solids or Moisture.

Preparation of the Dishes.

The nickel and aluminium dishes used for total solids and moisture determinations measure 3 ins. wide and 1 in. deep. The moisture determination dishes are fitted with lids made to " sit " inside them loosely and are not intended to be a tight fit. The dishes are so constructed that they can be handled with tongs without requiring the displacement of the lid at any time after the sample has been weighed into them. All total solids and moisture determinations should be carried out at 105°C. and adjustments should be made so that the temperature of the vacuum oven hot-plate is maintained at 105°C. Both dishes and lids have numbers stamped on their surfaces for ease of reference during a batch of determinations.

For moisture determinations the dishes are provided with lids, as it has been found by experience that greater accuracy is obtainable by their use.(10)

The dishes—dry, clean and empty—are placed in the " solids " vacuum oven operating at 105°C.

(10) " Official and Tentative Methods of Analysis of A.O.A.C.," 3rd Edit., 1930; *Ind. Eng. Chem.*, 1920, **12**, 40; *ibid.*, 1924, **16**, 741, 1163; *ibid.*, 1925, **17**, 311; *ibid.*, 1926, **18**, 272; *J. Assoc. Official Agric. Chem.*, 1923, **7**, 132; *ibid.*, 1925, **8**, 76, 301, 665; *ibid.*, 1926, **9**, 39, 40; *Cereal Chemistry*, 1924, **1**, 27; *ibid.*, 1925, **2**, 318; *ibid.*, 1926, **3**, 323.

The flanged door is securely closed and the vacuum is established in the total solids section. The dishes are allowed to remain in the vacuum oven for exactly 5 minutes, a period indicated by the alarm clock which is set and started immediately the vacuum gauge records a reading of 25 ins.

At the expiry of this period, the vacuum is discontinued, the dishes are transferred to the "solids" desiccator by means of the special tongs, the flanged door is closed securely and the dishes are allowed to remain in the desiccator for a period of five minutes for solids dishes and seven minutes for moisture dishes, the time again being determined by the alarm clock. Care must be taken to see that the cooling fluid is circulating freely by keeping the pump in operation.

The dishes are then removed one at a time and weighed, the operation being conducted as quickly as possible.

(1) Total Solids Determinations.

The sample is weighed into the "solids" dish prepared as described under "Preparation of Dishes" as quickly as possible by the method best suited to the substance, details of which appear subsequently under each substance.

The dishes are next transferred to the hot plate operating at 180°C. for milk products or 100°C. for other products, and are held in close contact with the plate by means of the *special depressor*. This ensures even heating over the whole surface of the dishes.

When the contents of the dishes are dry and very slight "browning" commences, the dishes are removed by the special tongs to the "solids" vacuum oven operating at 105°C. The door is securely closed and a vacuum of at least 25" is established. At this point the alarm clock is set for the required period of time.

At the expiry of this period, the vacuum is released; the dishes are transferred by the special tongs to the " solids " desiccator, the door of the desiccator is securely closed and the alarm clock is set for the required period, the pump circulating the cooling water being kept in operation. The dishes are then removed with the special tongs at the end of this period and weighed as rapidly as possible.

(2) Moisture Determinations.

The solid substance weighed into the dishes, prepared as described under " Preparation of Dishes," is spread evenly over the bottom surface and each dish is kept covered with its appropriate lid during the final weighing operations before the actual heating period.

The dishes covered with their respective lids are then transferred to the vacuum oven operating at 105 °C. and the flanged door is securely closed. A vacuum of at least 25″ is established and the alarm clock is set for the required period of time.

At the end of this period the vacuum is released, the dishes still covered by their appropriate lids are removed by the special tongs to the solids desiccator, the flanged door is securely closed and the alarm clock set again for the requisite time, the pump operating the cooling fluid being kept in operation.

After cooling, the dishes, with the lids still in position, are weighed as before.

B. General Outline of the Test for the Determination of Fat.

Principle of the Method.

The method employed is a modification of the well known Röse-Gottlieb method, i.e. hydrolysis with strong ammonium hydroxide, subsequent

extraction of the free fat by means of a mixture of ethyl ether and petroleum ether, and the final evaporation of the separated ether extract.

Reagents Required.

1. Distilled water.—A blank determination should show the supply to be free from matter extractable by ether and from any dissolved solids.

2. Ammonia.—This should be of A.R. quality conforming to the specifications associated with this designation.

3. Alcohol.—This should be 95% ethyl alcohol and should leave no residue upon evaporation.

4. Ethyl Ether.—This should be of A.R. quality conforming to the specifications associated with this designation.

5. Petroleum Ether.—This should be of A.R. quality, boiling point range 40-60°C.

It is strongly recommended that blank determinations be carried out fairly frequently using pure distilled water in place of the substance containing fat, and using the reagents in the manner and quantity detailed in the notes which follow.

Functions of the Reagents.

Distilled Water.—Water is added to concentrated milk products, and products of a similar nature, to reduce the fluidity to that approximately of ordinary whole milk. By this means also, it is attempted to produce a liquid capable of carrying the solids-not-fat in solution when they are dissolved in other reagents.

It may be necessary, at times, to raise the level of the aqueous layer in the fat extraction flask by the addition of water, in order to facilitate the removal of the major portion of the ethereal layer.

Ammonia.—The main objects of this reagent are (1) to dissolve the casein present in colloidal suspension in milk and milk products, and (2) to neutralise any free acids present. The viscosity

of the product is to some extent decreased and the fat is left available in a readily extractable form. It is said that phospholipoids are also destroyed if present.

ALCOHOL.—When ethyl ether is shaken vigorously with milk and milk products it tends to form what would appear to be an emulsion. It is found that the addition of alcohol breaks down this emulsion and also assists in the separation of the ethereal layer from the aqueous layer during the extractions.

ETHYL ETHER.—This reagent is the true fat extractant: it also dissolves small amounts of lactose and other solids-not-fat.

PETROLEUM ETHER.—The chief function of this reagent is not so much its use as a fat solvent as its ability in precipitating the traces of water invariably held in solution by ether: with the precipitation of this water the lactose and other solids-not-fat are also thrown out. The petroleum ether thereby purifies the ethyl ether extract and prevents erroneous results.

Note.—It is especially important that the specified amounts of reagents are strictly adhered to for it has been found by very careful check determinations that any deviation from the standard quantities results in error.(11)

(1) Preparation of the Dishes.

The aluminium dishes used for fat determinations measure $3\frac{1}{2}$ in. wide and 2 in. deep and no lids are necessary with their use.

The dishes—thoroughly cleaned, dried and empty—are placed in the "fats" vacuum oven operating at 135°C. The flanged door is securely closed and the vacuum is established as described in the section "A," p. 20.

(11) "The Technical Control of Dairy Products," Mojonnier and Troy. 2nd Edit., 1925, p. 47.

The dishes are allowed to remain in the vacuum oven for exactly five minutes, a period indicated by the alarm clock, which is started immediately the vacuum gauge records a reading of 25".

At the expiry of this period, the vacuum is discontinued and the dishes are transferred to the "fats" desiccator by means of the special tongs. The flanged door of the desiccator is closed securely and the dishes are allowed to remain in the desiccator for a period of seven minutes, the time again being determined by the alarm clock. Care must be taken to see that the cooling fluid is circulating freely by keeping the pump in operation.

The dishes are then removed, one at a time, for weighing, which operation is conducted as quickly as possible.

(2) Weighing of Sample.

(*a*) The method of weighing here described, which is essentially that of Mojonnier,[12] saves considerable time and loses nothing in accuracy if properly carried out and applies to liquids capable of being sucked up into pipettes with ease, such as whole milk, etc.

Figure 5 shows 1-gram, 2-gram and 5-gram pipettes respectively.

The method of weighing consists in deciding primarily the amount of sample required which will be selected according to the nature of the substance.

Having selected the quantity best suited to the substance, the requisite number of pipettes of the required capacity corresponding to the number of samples to be examined, are taken and filled with the respective samples by the normal process of sucking up the liquid to the mark etched on the neck. It is important that the samples should be

(12) "The Technical Control of Dairy Products," Mojonnier and Troy. 2nd Edit., 1925, p. 96, *et seq.*

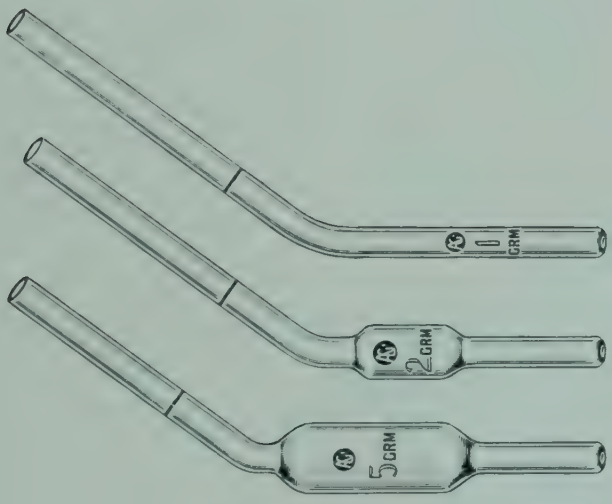

FIG. 5.

1 gram, 2 gram and 5 gram pipettes.

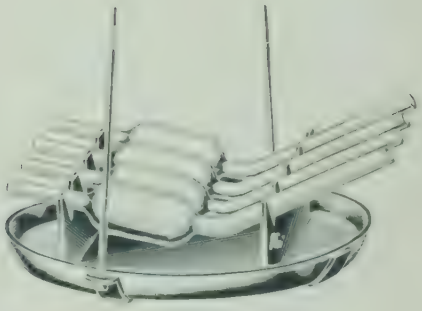

FIG. 6.

Special Rack Showing pipettes in position on the Balance Pan.

sucked up and allowed to flow out again completely at least once before finally adjusting to the mark.

As the pipettes are filled they are wiped on the outside free from adhering liquid and are placed as shown in figure No. 6 in a horizontal position in the metal cradle. This procedure is continued until all the samples have been so treated, after which the cradle and pipettes are placed bodily on the left-hand balance pan.

The total weight of the load is recorded and the first pipette is removed from the cradle and the contents discharged into the appropriate fat extraction flask, say No. 1. The pipette is allowed to drain for about 15 seconds, after which it is carefully withdrawn from the neck of the fat extraction flask and replaced on the cradle without loss of any drainings, after which a second weighing is made of the cradle with the full pipettes and the one empty pipette, and this weighing is recorded.

The difference between the first and second weights gives the weight of substance discharged into the fat extraction flask No. 1.

Pipette No. 2 is similarly removed from the cradle, and its contents carefully discharged into the special fat extraction flask, say No. 2, as just described: the pipette, as before, is drained for about 15 seconds, after which it is withdrawn, placed in the cradle together with the full pipettes and No. 1 empty pipette.

The cradle now contains the full pipettes and two empty pipettes, Nos. 1 and 2. The weight of the load is again recorded and subtracted from the second weight (i.e. cradle, plus full pipettes, plus one empty pipette, No. 1).

Again, this difference gives the weight of sample discharged into fat extraction flask No. 2.

This process is continued until all the samples in the pipettes have been so dealt with, after which

the cradle and empty pipettes are removed and the pipettes are set aside for cleaning (see separate section, p. 29, para. 4).

(*b*) The method here described is intended for use with substances which cannot be dealt with by means of the weighing pipettes described under (a).

To apply this method the flask (see Fig. No. 7) must be left in the balance case for a reasonable length of time, suspended from the balance stirrup hook by means of the metal collar shown in Fig. No. 8. The weight is recorded.

The sample under examination is then quickly weighed into the empty flask directly, care being taken that the flask is not handled unduly in order to prevent expulsion of air from the flask by expansion. This weight is also recorded and the difference between these weighings gives the weight of material taken.

(*c*) For materials which are not rapidly hygroscopic, e.g. butter, a small boat is provided (see Fig. No. 9). The boat is placed empty on the balance pan and left in the case for some minutes to attain equilibrium, after which its weight is obtained and recorded. The sample is placed in the boat, the weight again determined and recorded and the weight of sample taken is obtained by difference.

(3) Extraction of the Fat.

To the contents of the fat extraction flask add the reagents in the following order, carrying out each operation exactly as detailed below:—

First Extraction.

1. Add the requisite amount of distilled water from the water burette (if required—see instructions for individual substances). Cork and shake thoroughly (see Fig. 10).

2. Add the requisite quantity of ammonium hydroxide from the ammonium hydroxide burette

FIG. 7.

Special Fat Extraction Flask. Also shows relative positions of sample and extractants.

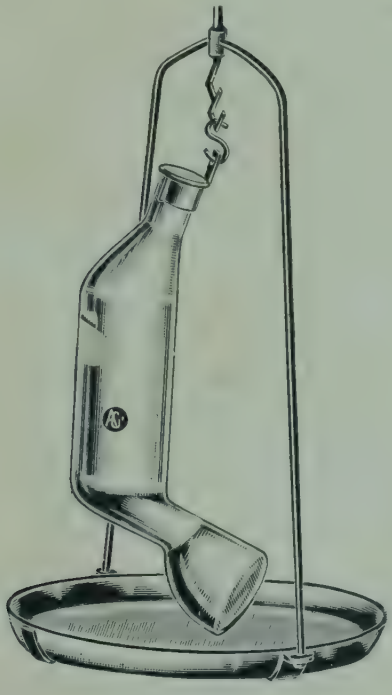

FIG. 8.

Fat Extraction Flask suspended from Balance stirrup by means of special metal collar.

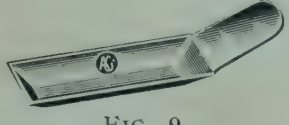

FIG. 9.

Butter Boat for use with materials not readily hygroscopic, etc.

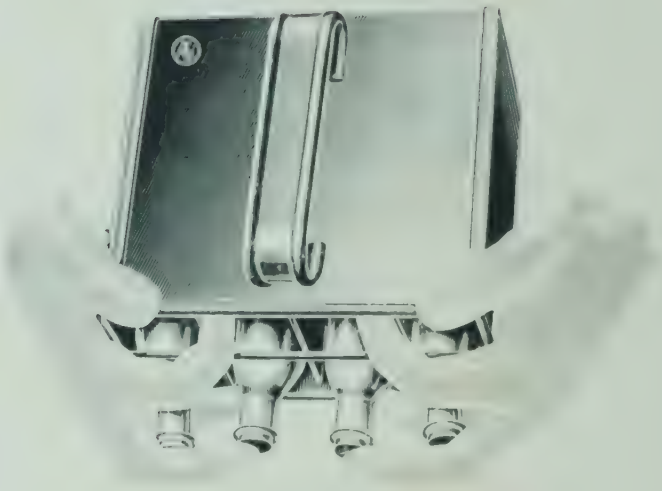

FIG. 10.

Showing Method of Shaking four Fat Extraction Flasks simultaneously in the centrifuge basket. Note position of operator's hands.

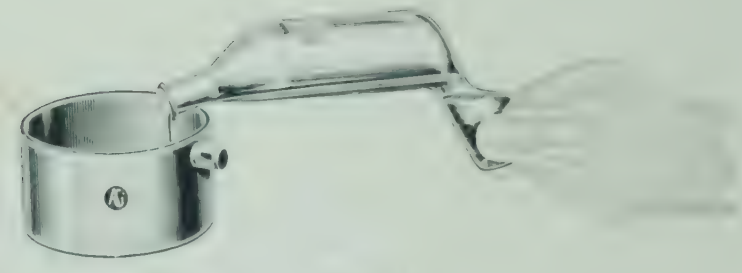

FIG. 11.

Showing Method of Decanting the mixed ethereal layer into the "fats" dish.

(see special section for amount required). Cork again, shake vigorously for about 20 seconds.

3. Add the requisite amount of alcohol from the alcohol burette (see appropriate section for amount required). Cork and shake vigorously for 30 seconds.

4. Add the requisite quantity of ethyl ether from the ethyl ether burette (see appropriate section for amount required). Cork and shake vigorously for 20 seconds.

5. Add the requisite quantity of petroleum ether from the petroleum ether burette (see appropriate section for amount required). Cork and shake vigorously for 20 seconds.

The flask is placed, tightly corked, in the centrifuge basket and whirled for 30 seconds. It is then removed and the dividing line between the aqueous and ethereal layers is adjusted by the addition of distilled water to such a point in the constricted neck of the flask that the aqueous layer level lies just below the level of the horizontal side of the flask during the pouring operations (see Fig. No. 7).

After the aqueous level has been so adjusted, the flask is tilted as shown in Fig. No. 11 and the major portion of the ethereal layer is decanted into the appropriate "fats" dish, which is then immediately placed on the "fats" hot plate operating at 135°C., the glass hood is drawn over it and the suction pump is set in operation.

The decanting operation can, if the aqueous level is properly adjusted, be carried out in such a manner that only a few drops of ether remain in the flask.

SECOND EXTRACTION.

(1) Add *no* water.

(2) Add *no* ammonium hydroxide.

(3) Add the requisite quantity of alcohol from

the alcohol burette (see appropriate section for amount required). Cork and shake vigorously for 20 seconds.

(4) Add the requisite amount of ethyl ether from the ethyl ether burette (see appropriate section for amount required). Cork and shake vigorously for 20 seconds.

(5) Add the requisite amount of petroleum ether from the petroleum ether burette (see appropriate section for amount required). Cork and shake vigorously for 20 seconds.

Place the flask in the basket and centrifuge for 30 seconds. If necessary adjust the aqueous level and decant the ethereal layer into the same "fats" dish as described under "First Extraction," p. 27, para. 5.

Care must be taken to see that the "fats" dish is cold, or at best only faintly warm, before the second ethereal extraction is decanted.

The ethers are evaporated as described under "First Extraction," p. 27, para. 5.

(4) Treatment of Dish and Fat.

After all the ether has been expelled by evaporation on the hot plate the dish is placed in the "fats" vacuum oven operating at 135°C., the flanged door is securely closed, the vacuum is established and the dish is heated for 5 minutes, the alarm clock being set in operation when the gauge registers 25".

After the expiry of this period the dish is removed by means of the special tongs and cooled in the "fats" desiccator for seven minutes, the period again being timed by the alarm clock.

The dish is then weighed rapidly and the weight recorded.

The difference between the weight of the dish plus fat, and the weight of the dish, gives the weight of fat in the amount of sample taken.

From this the percentage fat content is calculated.

Precautions to be observed for the obtaining of accurate results for Fats and Total Solids Determinations.

1. The temperatures of the desiccators and of the inside of the balance case should be the same.

2. The desiccant in the desiccators should be checked frequently.

3. On no account whatever must fat from previous determinations be allowed to remain in a fat dish, and ethereal extracts from a subsequent estimation be poured on to the fatty residue. All dishes, both solids and fats, must be scrupulously cleaned with soap and hot water, rinsed free from soap, etc., with distilled water, hand-dried with a clean cloth and returned to the respective desiccator.

4. All glassware should be cleaned after each estimation with soap and hot water and then subsequently rinsed with distilled water and dried by means of hot air.

At the end of a day's work all glassware should be filled with chromic acid, emptied the following morning, and washed in distilled water and dried by means of hot air.

5. Any deformation of the moisture or "fats" dishes should be "worked out" by placing the dish in question on a small marble slab or a piece of plate glass and rubbing the deformed portion firmly with the fingers.

The dishes should be examined critically by the operator every time they are used: especial attention is directed to any bulging or dents on the bottoms of the dishes.

This is extremely important.

6. In decanting the ethereal layer from the fat extraction flask, the operator should regulate the flow of ether to a slow stream at first, gradually increasing the speed.

7. It is essential that all glass apparatus used in the test is perfectly clean and *dry*.

8. Only the best corks must be used for the fat extraction flasks. Old and worn or faulty corks should be discarded immediately.

9. The vacuum ovens and hot plates must operate at the temperatures recommended and these should be checked fairly frequently.

10. Great care must be exercised to see that ethereal extracts are not poured into hot "fats" dishes.

11. All times recommended for heating, cooling, shaking, etc., should be rigidly observed.

Summary of the Sequence of Operations Required for the Complete Total Solids and Fats Test on Liquid Products.

It is assumed that the tester has been started up in accordance with the instructions under "Instructions for Starting up the Tester" section, p. 18.

1. Heat the respective "fats" and "solids" dishes, empty and clean, in the respective vacuum ovens for exactly five minutes (timed by the alarm clock) with the gauge showing a reading of 25", at least.

2. Release the vacuum and transfer the dishes to their respective desiccators, keeping the pump in operation. Cool for exactly five minutes in the case of the solids and seven minutes in the case of the "fats" dishes, timing again by the alarm clock.

3. Firstly, weigh the "solids" dishes and record the weights and the dish numbers, and then replace them in the "solids" desiccator.

4. Next weigh the "fats" dishes alone, record their weights and numbers and replace them in the cooling oven.

5. Weigh, as described, the requisite amount of sample in the appropriate pipette and transfer the weighed amount to a fat extraction flask as described in Section B (2), p. 24.

6. Weigh the required amount of sample by means of the appropriate pipette, as described in section B (2), p. 24, and transfer the pipetted quantity to a "solids" dish. Add sufficient water, if necessary, to make the volume up to about 2 ml. Mix thoroughly and place the dishes on the hot plate.

7. Evaporate the material in the "solids" dishes to dryness, effectively establishing contact between dish and hot plate by means of the depressor. (Section A (1), p. 20).

8. When dry, transfer the "solids" dish rapidly to the vacuum oven and heat at the correct reduced pressure for the required period as timed by the alarm clock.

9. During this period carry out the first fat extraction as described in section B (3), p. 26.

10. After the completion of the first extraction, carry out the second fat extraction as described in section B (3), p. 27.

11. About this point the total-solids alarm-bell should ring: transfer the "solids" dishes to the "solids" desiccator and set the alarm clock for the required period.

12. The ether should, by this time, have evaporated completely: transfer the "fats" dishes to the "fats" vacuum oven and set the alarm clock for the required period.

13. The period of cooling of the "solids" dishes should by this time have elapsed and the alarm bell should ring; remove the dishes one at a time as rapidly as possible and weigh. Record the weights.

14. The "fats" dishes should by this time be practically ready for withdrawal and when the "fats" alarm clock rings, transfer the dishes to the desiccator, and set the alarm clock for the required period.

15. When the "fats" alarm clock rings, withdraw the dishes as rapidly as possible one at a time and weigh. Record the weights.

16. Calculate the percentage total solids and fat in the sample.

In the preceding pages an endeavour has been made to give a comprehensive survey of the "Technico" Test Unit together with the general principles involved and the outline of the methods in use.

The remarks concerning the total solids test and "fats" test, although of a purely general character, require only superficial modifications to suit individual cases and in all the essential details the methods outlined are generally applicable.

In the pages which follow as full a description as possible has been appended of the details of the total solids and "fats" determinations on a very large number of foodstuffs, details which have been tested over a long period and have been found to give reliable results.

CHAPTER II.

MILK (Includes Fresh Milk, Skim Milk, Whey and Buttermilk).

Method of Sampling of Fresh Milk, Skim Milk and Buttermilk.

In large dairies where milk is produced on the spot, special methods applicable only to the apparatus and method of handling employed will doubtless operate and no directions given here would be of any value.

In depots or in factories purchasing and receiving deliveries of milk in churns, the following simple method has been found quite suitable:—

The churns are firstly labelled distinctly for reference purposes.

At least 10% of the delivery should be selected. The tops of the selected churns are removed as required, and not before, and the contents of each churn thoroughly mixed by inserting a "milk plunger" and plunging at least thirty times before taking the sample, which is conveniently abstracted while the product is still in motion by means of an aluminium jug with a spout.

Each sample should be placed in a clean and dry screw-cap jar labelled appropriately with the mark corresponding to that on the churn from which it was taken.

The sample, so taken, should be stored in a refrigerator until required for analysis, but on no account should the product be allowed to freeze.

Method of Sampling of Whey.

Owing to the ease and rapidity with which fat and casein separate from whey, some modifications in the method of sampling are necessary.

The most satisfactory method consists in abstracting small measured amounts from the mass to be tested immediately after mixing, and transferring these measured amounts directly to the vessel in which the test is to be carried out. By this means fat separation is reduced to a minimum: it has been found that fat once separated from whey is re-incorporated only with the greatest difficulty and for this reason sampling should be carried out as soon as the whey has been produced.

Total Solids Determination.

Approximately two grams of the well mixed sample are weighed into the prepared total solids dish ("Preparation of the Dishes," p. 19) using the 2-gram pipette in conjunction with the special pipette rack (section B (2), p. 25). No water must be added and the sample should be spread evenly over the entire surface of the dish by gentle rotation, the dish being held by the special tongs.

The dish should then be placed on the "solids" hot plate operating at 180°C. and the contents evaporated to apparent dryness using the dish depressor as described in section A (1), p. 20.

The dish is then transferred to the "solids" vacuum oven operating at 105°C., and the alarm clock is set for ten minutes from the time the gauge shows a reading of 25″.

At the end of this period, the dish is transferred to the "solids" desiccator and the alarm clock is again set for five minutes, taking especial care that the cooling fluid is flowing freely.

At the end of the cooling period, the dish is transferred to the balance and weighed rapidly.

Note.—Following a suggestion made by Revis[13], Bolton conducted a series of tests in which small quantities of acetone were added to the dishes before the evaporation of the milk.

As a result of these tests he claims great rapidity and accuracy.[14]

Fat Determination.

Weigh approximately 10 grams of the well-mixed sample using the 10-gram pipette in conjunction with the special pipette rack (sec. B (2), p. 25). In transferring the contents of the pipette to the fat extraction flask, allow the pipette to drain for 15 seconds after the sample has been discharged: the last drop adhering to the inside of the pipette is finally blown out into the flask.

No water must be added to the sample.

First Extraction.

(1) Add 1.5 ml. ammonium hydroxide—cork and shake thoroughly.
Then (2) Add 10 ml. of alcohol—cork and shake 30 seconds.
,, (3) Add 25 ml. of ethyl ether—cork and shake for 20 secs.
,, (4) Add 25 ml. of petroleum ether—cork and shake for 20 seconds.

Keep the fat extraction flask securely corked and centrifuge for 30 seconds, the whole operation being completed in 30 turns of the centrifuge handle. Uncork the flask carefully and decant the ethereal layer into the prepared "fats" dish (sec. B (3), p. 27, "First Extraction").

Second Extraction.

(1) Add *no* water.
(2) Add *no* ammonium hydroxide.
(3) Add 5 ml. alcohol—cork and shake for 20 seconds.

(13) *Analyst*, 1907, **32**, 284.
(14) *Ibid.*, 1924, **49**, 419.

Then (4) Add 15 ml. of ethyl ether—cork and shake for 20 seconds.

,, (5) Add 15 ml. of petroleum ether—cork and shake for 20 seconds.

Keep the fat extraction flask securely corked and centrifuge for 30 seconds as before, the whole operation being completed in 30 turns of the centrifuge handle.

If, as pointed out in section B (3), p. 27, "First Extraction," the line of separation of the liquids is too low, distilled water may be added just before the ethereal layer is poured into the "fats" dish during the second extraction.

Remove the cork of the fat extraction flask carefully and decant the ethereal layer into the prepared "fats" dish as in the first extraction (sec. B (3), p. 27, "First Extraction").

After the evaporation of the ethers (sec. B (3), p. 27) the dish is heated in the "fats" vacuum oven operating at 135°C. for five minutes, timed by the alarm clock, after the gauge registers 25″.

After the expiry of this period the dish is removed and transferred to the "fats" desiccator, and the alarm clock is again set for seven minutes, especial care being taken to see that the cooling fluid is flowing freely.

When the dish has been cooled for the requisite period it is transferred to the balance and weighed rapidly.

CREAM.

Method of Sampling.

As in the case of milk, large creameries will doubtless employ special procedure applicable to some particular method of production and handling and the remarks which follow will find no application to these special cases.

In depots or in factories receiving and purchasing deliveries of cream in churns the following simple method gives satisfactory results:—

The churns should first be labelled distinctly for reference purposes.

At least 10% of the delivery should be selected, the tops of the selected churns removed as required, and not before, and the contents of each churn thoroughly mixed by inserting a "milk-plunger" and plunging vigorously at least twenty times, making sure that the plunger reaches the bottom of the churn each time.

The samples are abstracted whilst the cream is still in motion, by means of an aluminium jug with a spout, and are transferred to their appropriate screw-cap sample jars suitably labelled with the marks of the corresponding churns.

The samples so taken should be stored in a refrigerator until required for analysis. On no account should the cream be allowed to freeze.

Total Solids Determination.

Weigh approximately 1 gram or 0.5 gram of the well mixed sample into the prepared total solids dish ("Preparation of Dishes," p. 19), using the 2-gram pipette in conjunction with the special pipette cradle (sec. B (2), p. 25).

In the case of some creams it may be extremely difficult to manipulate the sample by means of the pipette: in such instances it is advisable to weigh the sample directly into the total solids dish, or to weigh from the butter boat described in section B (2c), p. 26.

In cases where it is known that the cream contains less than 25% of fat the amount of sample taken should be 1 gram, using the 1-gram pipette, or either of the alternative methods outlined above.

In cases where it is known that the cream contains more than 25% of fat the amount of sample should be reduced to 0.5 gram. As before, one of the alternative methods can be employed.

For cream containing less than 25% fat, add 1 ml. distilled water to the sample in the "solids" dish.

For cream containing more than 25% fat, add 1.5 ml. distilled water to the sample in the "solids" dish.

The sample should be spread evenly over the bottom of the dish by gentle rotation, the dish being held by the special tongs.

The dish is then placed on the "solids" hot plate operating at 180°C. and the contents are evaporated to apparent dryness using the dish depressor as described in section A (1), p. 20.

It is then transferred to the "solids" vacuum oven operating at 105°C. and the alarm clock is set for ten minutes from the time the gauge shows a reading of 25".

At the end of this period, the dish is transferred to the "solids" desiccator and the alarm clock is again set for five minutes, especial care being taken to see that the cooling fluid is flowing freely.

At the end of the cooling period, the dish is transferred to the balance and weighed rapidly.

Fat Determination.

If the nature of the cream permits, the pipette method of weighing out the sample in conjunction with the pipette cradle may be adopted (sec. B (2), p. 25), using the 2-gram or 1-gram pipette as necessary. In transferring the contents of the pipette to the fat extraction flask allow the pipette to drain for 20 seconds after the sample has been discharged: the last drop adhering to the inside of the pipette is finally blown out into the flask.

If this is not possible then the butter boat may be used (sec. B (2c), p. 26) for the weighing operation or the sample can be weighed directly into the fat flask (sec. B (2b), p. 26).

For cream containing less than 25% fat use approximately 2 grams weighed accurately of the well mixed sample.

For cream containing more than 25% fat use approximately 1 gram weighed accurately of the well mixed sample.

FIRST EXTRACTION.

(1) Add 5 ml. distilled water for cream containing less than 25% fat—cork and shake thoroughly.
Add 6 ml. distilled water for cream containing more than 25% fat—cork and shake thoroughly.

Then (2) Add 1.5 ml. ammonium hydroxide—cork and shake thoroughly.

,, (3) Add 10 ml. alcohol—cork and shake for 30 seconds.

,, (4) Add 25 ml. ethyl ether—cork and shake for 20 seconds.

,, (5) Add 25 ml. of petroleum ether—cork and shake for 20 seconds.

Keep the fat extraction flask securely corked and centrifuge for 30 seconds, the whole operation being completed in 30 turns of the centrifuge handle. Uncork and decant the ethereal layer into the prepared "fats" dish (sec. B (3), p. 27, "First Extraction").

SECOND EXTRACTION.

(1) Add *no* distilled water.

(2) Add *no* ammonium hydroxide.

(3) Add 5 ml. alcohol—cork and shake for 20 seconds.

Then (4) Add 25 ml. of ethyl ether—cork and shake for 20 seconds.

,, (5) Add 25 ml. of petroleum ether—cork and shake for 20 seconds.

Keep the fat extraction flask securely corked and centrifuge for 30 seconds, the whole operation being completed in 30 turns of the centrifuge handle.

If, as pointed out in sec. B (3), p. 27, the line of separation of the two liquids is too low distilled water may be added just before the ethereal layer is poured off into the "fats" dish during the second extraction. Uncork the flask carefully and decant the ethereal layer into the prepared "fats" dish as in the first extraction (sec. B (3), p. 27, "First Extraction").

After the evaporation of the ethers (sec. B (3), p. 27) the dish is heated in the "fats" vacuum oven at 135°C. for five minutes, timed by the alarm clock, from the time the gauge shows 25″.

After the expiry of this period the dish is removed and transferred to the "fats" desiccator and the alarm clock is again set for seven minutes, especial care being taken to see that the cooling fluid is flowing freely.

When the dish has been cooled for the requisite period it is transferred to the balance and weighed rapidly.

UNSWEETENED CONDENSED MILK (Includes Evaporated Milks and Condensed Buttermilk).

Method of Sampling.

The method outlined here applies to the products in tins. Factories producing the commodities will doubtless possess special methods of sampling applicable to their method of manufacture and handling.

Drain the contents of the tin into a large shallow vessel and "work" the sample well with a pestle, using both horizontal and vertical motions until the sample is thoroughly homogeneous.

Thorough mixing is facilitated by slightly warming the sample should it appear to be too viscous

to stir adequately. The sample should be stored in a screw-cap jar in a cool place until required for analysis.

In any work on products of the nature here described, the greatest possible care should be exercised in sampling from bulk and the sample should always be thoroughly mixed before any work is carried out.

This is of extreme importance.

Total Solids Determination.

Weigh accurately about 1 gram of the well mixed sample, except in the case of condensed buttermilk, when 0.5 gram is used.

Use the 1-gram pipette in conjunction with the pipette cradle (sec. B (2), p. 25) or weigh the sample directly into the prepared total solids dish ("Preparation of the Dish," p. 19).

To the sample in the dish add 1 ml. distilled water in all cases except that of condensed buttermilk, when 2 ml. distilled water should be added.

Mix the sample and the water together thoroughly in the dish, making sure that the well-mixed sample is spread evenly over the entire bottom surface of the dish by gentle rotation, the dish being held by the special tongs.

The dish should then be placed on the "solids" hot plate operating at 180°C. and the contents should be evaporated to apparent dryness, using the dish depressor as described in sec. A (1), p. 20.

The dish is then transferred to the "solids" vacuum oven operating at 105°C., and the alarm clock is set for ten minutes from the time the gauge shows a reading of 25″.

At the end of this period transfer the dish to the "solids" desiccator and again set the alarm clock for five minutes, taking especial care that the cooling fluid is flowing freely.

At the end of the cooling period transfer the dish to the balance and weigh rapidly.

Fat Determination.

In any work on products of the nature here described, the greatest possible care should be exercised in sampling from bulk and the sample should always be thoroughly mixed before any work is carried out. *This is of extreme importance.*

If the nature of the product permits the pipette method of weighing out the sample in conjunction with the pipette cradle may be adopted (sec. B (2), p. 25). If this is not possible the sample may be weighed directly into the fat extraction flask (sec. B (2*b*), p. 26).

Weigh 5 grams of the well mixed sample either in the 5-gram pipette or directly into the fat extraction flask: in the case of condensed buttermilk, 3 grams of sample should be taken. Transfer to the flask in the usual way (sec. B (2*a*), p. 25).

First Extraction.

(1) Add 4 ml. distilled water—cork and shake thoroughly.
Add 6 ml. distilled water, in the case of condensed buttermilk—cork and shake thoroughly.

Then (2) Add 1.5 ml. ammonium hydroxide—cork and shake thoroughly.

,, (3) Add 10 ml. alcohol—cork and shake thoroughly for 30 seconds.

,, (4) Add 25 ml. ethyl ether—cork and shake thoroughly for 20 seconds.

,, (5) Add 25 ml. petroleum ether—cork and shake thoroughly for 20 seconds.

Keep the fat extraction flask securely corked and centrifuge for 30 seconds, the whole operation being completed in 30 turns of the centrifuge handle.

Uncork the flask carefully and decant the ethereal layer into the prepared "fats" dish (sec B (3), p. 27, "First Extraction").

SECOND EXTRACTION.

(1) Add *no* distilled water.

(2) Add *no* ammonium hydroxide.

(3) Add 5 ml. alcohol—cork and shake for 20 seconds.

Then (4) Add 25 ml. ethyl ether—cork and shake for 20 seconds.

,, (5) Add 25 ml. petroleum ether—cork and shake for 20 seconds.

Note.—In the case of condensed buttermilk only 15 ml. of each of the ethers should be used in this second extraction.

Keep the fat extraction flask securely corked and centrifuge for 30 seconds as before, the whole operation being completed in 30 turns of the centrifuge handle.

Uncork the "fats" flask carefully and decant the ethereal layer into the prepared "fats" dish as in the first extraction (sec. B (3), p. 27, "First Extraction").

If, as pointed out in sec. B (3), p. 27, "First Extraction," the line of separation of the liquids is too low, distilled water may be added just before the ethereal layer is poured off into the "fats" dish during the second extraction.

After the evaporation of the ethers (sec. B (3), p. 27) heat the "fats" dish in the "fats" vacuum oven, operating at 135°C., setting the alarm clock for five minutes from the time the gauge shows a reading of 25".

After the expiry of this period remove the dish to the "fats" desiccator and again set the alarm clock for seven minutes, taking especial care to see that the cooling fluid is flowing freely.

When the dish has cooled for the requisite period transfer it to the balance and weigh rapidly.

SWEETENED CONDENSED MILK.

Method of Sampling.

The method outlined here applies to the products in tins.

Factories producing the commodities will doubtless possess special methods of sampling applicable to their method of manufacture and handling.

Drain the contents of the tin or container into a large shallow vessel, and " work " the sample well with a pestle, using both horizontal and vertical motions until the sample is thoroughly homogeneous.

Should any separation of crystals have occurred, the milk should be warmed gently in order to disperse them before sampling.

The sample taken should be stored in a screw-cap sample jar in a cool place until it is required for analysis.

Total Solids Determination.

In any work on products of this nature the greatest possible care should be taken in sampling from bulk and the sample should always be thoroughly mixed before any work is carried out.

This is of extreme importance as the constituents are likely to separate to some extent on standing.

If the nature of the product permits the pipette method of weighing the sample in conjunction with the pipette cradle may be adopted. If this is not possible the sample may be weighed directly into the prepared total solids dish.

In delivering the sample to the dish the operator should place the sample in drops over various parts of the bottom of the dish in order that the sample may the more readily be dispersed when the water is added.

If the pipette method of weighing is adopted, use the 1-gram pipette. Weigh out approximately

0.25 gram of well mixed sample into the prepared "solids" dish ("Preparation of the Dish," p. 19). Add 2 ml. of hot distilled water[13, 14] and rotate the dish using the special tongs until complete dispersion has taken place: in the case of sweetened condensed milk this is somewhat slow. Make sure the sample is homogeneous and evenly spread over the entire bottom surface of the dish.

The dish should then be placed on the "solids" hot plate operating at 180°C. and the contents should be evaporated to apparent dryness, using the dish depressor as described in sec. A (1), p. 20.

Transfer the dish to the "solids" vacuum oven operating at 105°C. and set the alarm clock for 90 minutes from the time the gauge shows a reading of 25″.

Some authorities claim that if results are needed in the minimum amount of time, the sample should be heated in the "solids" vacuum oven for 20 minutes at a gauge reading of not less than 20″ subsequently subtracting 0.3% from the final figure obtained: these workers claim results almost identical with those obtained by the 90 minutes method. For further information the paper by Lampitt, Hughes and Bogod[15] should be consulted.

Whichever method has been used, transfer the dish at the end of the heating period to the "solids" desiccator and again set the alarm clock for five minutes, taking especial care that the cooling fluid is flowing freely.

At the end of the cooling period transfer the dish to the balance and weigh rapidly.

Fat Determination.

In any work on products of this nature the greatest possible care should be exercised in sampling from bulk and the sample should always

(13) *Loc. cit.*
(14) *Loc. cit.*
(15) *Analyst*, 1924, **49**, 414.

be thoroughly mixed before any work is carried out. *This is of extreme importance as the constituents are likely to separate to some extent on standing.*

The pipette method in conjunction with the pipette cradle should be used (sec. B (2), p. 25).

Use the five-gram pipette and weigh approximately 5 grams of the well-mixed sample. Transfer to the fat extraction flask in the usual way (sec. B (2), p. 25).

First Extraction.

(1) Add 8 ml. distilled water—cork and shake very thoroughly.

Then (2) Add 1.5 ml. ammonium hydroxide—cork and shake very thoroughly.

,, (3) Add 10 ml. alcohol—cork and shake for 60 seconds.

,, (4) Add 25 ml. ethyl ether—cork and shake for 60 seconds.

,, (5) Add 25 ml. petroleum ether—cork and shake for 60 seconds.

Keep the fat extraction flask securely corked and centrifuge for 60 seconds, the whole operation being completed in 60 turns of the centrifuge handle.

Uncork the flask carefully and decant the ethereal layer into the prepared "fats" dish (sec. B (3), p. 27, "First Extraction").

Second Extraction.

(1) Add *no* distilled water.

(2) Add *no* ammonium hydroxide.

(3) Add 5 ml. alcohol—cork and shake for 20 seconds.

Then (4) Add 25 ml. of ethyl ether—cork and shake for 20 seconds.

,, (5) Add 25 ml. of petroleum ether—cork and shake for 20 seconds.

Keep the fat extraction flask securely corked and centrifuge for 60 seconds as before, the whole operation being completed in 60 turns of the centrifuge handle.

Uncork the fat extraction flask carefully and decant the ethereal layer into the prepared "fats" dish as in the first extraction (sec. B (3), p. 27, "First Extraction").

If, as pointed out in sec. B (3), p. 27, "First Extraction," the line of separation of the liquids is too low, distilled water may be added just before the ethereal layer is poured off into the "fats" dish during the second extraction.

After the evaporation of the ethers (sec. B (3), p. 27) heat the "fats" dish in the "fats" vacuum oven operating at 135°C., setting the alarm clock for five minutes from the time the gauge shows a reading of 25".

After the expiry of this period, remove the dish to the "fats" desiccator and again set the alarm clock for seven minutes, taking especial care to see that the cooling fluid is flowing freely.

When the dish has cooled for the requisite period, transfer it to the balance and weigh rapidly.

ICE CREAM MIX.

Method of Sampling.

In most cases where analysis of ice cream mix becomes necessary for purposes of standardisation, the chemist is usually called upon to take his sample immediately upon completion of the mixing operations. In this event the mix is usually perfectly homogeneous and has not remained undisturbed in the mixing tanks for any appreciable length of time. In this case, after running the mixing mechanism in the tanks for about one minute, a sample can be withdrawn into a screw-cap sample

jar and placed in a refrigerator until required for analysis: on no account must the sample be allowed to freeze.

In cases where the homogeniety of the product is open to question owing to standing or to some other cause, the "mix" should be remixed for at least 15 minutes before a sample is removed.

Total Solids Determination.

The sample may be weighed by means of the pipette method (sec. B (2), p. 25) in conjunction with the pipette cradle, in which case the 1-gram pipette is used; or, if preferred, it may be weighed directly into the prepared "solids" dish ("Preparation of the Dish," p. 19).

Whichever method, however, is adopted, 1 gram of the well-mixed sample is taken for the determination. Add 1 ml. of distilled water to the sample in the dish, mix thoroughly, and spread the sample evenly over the bottom of the dish by gentle rotation of the dish and contents with the special tongs.

Place the dish on the "solids" hot plate operating at 180°C. and evaporate the contents to apparent dryness using the dish depressor as described in section A (1), p. 20.

It is then transferred to the "solids" vacuum oven operating at 105°C., and the alarm clock is set for ten minutes from the time the gauge shows a reading of 25″.

At the end of this period transfer the dish to the "solids" desiccator, and again set the alarm clock for five minutes, taking especial care to see that the cooling fluid is flowing freely.

At the end of the cooling period transfer the dish to the balance and weigh rapidly.

Fat Determination.

In weighing the sample, the pipette in conjunction with the pipette cradle can be used (sec. B (2), p. 25), or, if preferred, the sample can be weighed

directly into the "fats" flask (sec. B (2*b*), p. 26). If the pipette method is used, the 5-gram pipette should be employed.

Take 5 grams of the well-mixed sample for the determination. In transferring the contents of the pipette to fat extraction flask, allow the pipette to drain for 15 seconds after the sample has been discharged: the last drop adhering to the inside of the pipette is finally blown out into the flask.

First Extraction.

(1) Add 5 ml. of distilled water—cork and shake thoroughly.

Then (2) Add 1.5 ml. of ammonium hydroxide—cork and shake thoroughly.

,, (3) Add 10 ml. of alcohol—cork and shake for 30 seconds.

,, (4) Add 25 ml. of ethyl ether—cork and shake for 30 seconds.

,, (5) Add 25 ml. of petroleum ether—cork and shake for 30 seconds.

Keep the fat extraction flasks securely corked and centrifuge for 30 seconds, the whole operation being completed in 30 turns of the centrifuge handle.

Uncork the flask carefully and decant the ethereal layer into the prepared "fats" dish. Evaporate the ethers. (Sec. B (3), p. 27, "First Extraction").

Second Extraction.

(1) Add *no* distilled water.

(2) Add *no* ammonium hydroxide.

(3) Add 5 ml. alcohol—cork and shake for 20 seconds.

Then (4) Add 25 ml. of ethyl ether—cork and shake for 20 seconds.

,, (5) Add 25 ml. of petroleum ether—cork and shake for 20 seconds.

Keep the fat extraction flask corked and centrifuge for 30 seconds, the whole operation being completed in 30 turns of the centrifuge handle.

Uncork the fat extraction flask carefully and decant the ethereal layer into the prepared "fats" dish as in the first extraction. Evaporate the ethers. (Sec. B (3), p. 27, "First Extraction").

If, as pointed out in sec. B (3), p. 27, "First Extraction," the line of separation of the liquids is too low, distilled water may be added just before the ethereal layer is poured off into the "fats" dish during the second extraction.

After the evaporation of the ethers (sec. B (3), p. 27) heat the "fats" dish in the "fats" vacuum oven operating at 135°C., setting the alarm clock for five minutes from the time the gauge shows a reading of 25″.

After the expiry of this period, remove the dish to the "fats" desiccator and again set the alarm clock for seven minutes, taking especial care to see that the cooling fluid is flowing freely.

When the dish has cooled for the requisite period, transfer it to the balance and weigh rapidly.

BUTTER.

Method of Sampling.

Owing to the heterogeneous nature of butter, sampling is a matter of extreme difficulty.

Butter in the Churn.

Remove with a spatula about a dozen $\frac{1}{4}$ oz. pieces from various parts of the churn and store after mixing thoroughly in screw-cap jars and place them in a refrigerator until required for use.

Butter in Tubs.

Samples of butter are removed by means of a "trier" from the edge, from a point half-way between this point and the centre and lastly from

the middle of the block of butter. The samples are placed together in a screw-capped sample jar and thoroughly mixed and kept in a refrigerator until required for use.

Total Solids Determination.

Weigh accurately about 1 gram of the sample directly into the prepared "solids" dish ("Preparation of the Dish," p. 19). Add no water to the sample in the dish.

Place the dish on the "solids" hot plate operating at 180°C. and keep it there until spattering ceases or until the first signs of a brown discoloration appear, using the dish depressor as described in sec. A (1), p. 20.

At this point transfer the dish to the "solids" vacuum oven operating at 105°C. and set the alarm clock for ten minutes from the time the gauge shows a reading of 25″.

After the expiry of this period transfer the dish to the "solids" desiccator, again setting the alarm clock for five minutes, taking especial care to see that the cooling fluid is flowing freely.

After the dish has cooled for the requisite period, transfer it to the balance and weigh rapidly.

Fat Determination.

The sample may be weighed out either by means of the weighing boat (sec. B (2*c*), p. 26) or directly into the fats extraction flask (sec. B (2*b*), p. 26).

Take 1 gram of the sample for the determination.

First Extraction.

(1) Add 8 ml. of hot distilled water—cork and shake thoroughly.

Then (2) Add 1.5 ml. ammonium hydroxide—cork and shake thoroughly.

,, (3) Add 10 ml. of alcohol—cork and shake for 30 seconds.

Then (4) Add 25 ml. of ethyl ether—cork and shake for 20 seconds.

,, (5) Add 25 ml. of petroleum ether—cork and shake for 20 seconds.

Keep the fat extraction flask securely corked and centrifuge for 30 seconds, the whole operation being completed in 30 turns of the centrifuge handle.

Uncork the flask carefully and decant the ethereal layer into the prepared "fats" dish. Evaporate the ethers. Sec. B (3), p. 27, "First Extraction").

Second Extraction.

(1) Add *no* distilled water.

(2) Add *no* ammonium hydroxide.

(3) Add 5 ml. alcohol—cork and shake for 20 seconds.

Then (4) Add 25 ml. ethyl ether—cork and shake for 20 seconds.

,, (5) Add 25 ml. petroleum ether—cork and shake for 20 seconds.

Keep the fat extraction flask securely corked and centrifuge for 30 seconds, the whole operation being completed in 30 turns of the centrifuge handle.

Uncork the flask carefully and again decant the ethereal layer into the prepared "fats" dish. Evaporate the ethers.

If, as pointed out in sec. B (3), p. 27, "First Extraction," the line of separation of the liquids is too low, distilled water may be added just before the ethereal layer is poured off into the prepared "fats" dish during the second extraction.

After the evaporation of the ethers, heat the "fats" dish in the "fats" vacuum oven operating at 135°C., setting the alarm clock for five minutes from the time the gauge shows a reading of 25".

After the expiry of this period, remove the dish to the "fats" desiccator and again set the alarm clock for seven minutes, taking especial care to see that the cooling fluid is flowing freely.

When the dish has cooled for the requisite period transfer it to the balance and weigh rapidly.

CHEESE.

Method of Sampling. (16)

Cut a wedge-shaped piece extending from the edges of the cheese to the centre. Cut this wedge into strips and pass the strips through a mincing machine three times. The resulting sample is then ready for analysis.

If this form of sampling is not possible, sampling should be carried out by means of a steel butter sampler. The sampler should be inserted at a point situated about half-way between the edge and the centre of the cheese and extending either completely through the cheese or at least half-way through it. Two other samples should be drawn similarly at points situated at the centre and as near as possible to the edge.

Pass the three plugs of cheese so obtained through a mincer three times and use the sample so prepared for the analysis required.

Total Solids Determination.

Weigh accurately about 0.5 gram of the prepared sample in the prepared "solids" dish. A small blunt glass rod should be weighed with the sample and should have undergone preparation with the empty dish ("Preparation of the Dish," p. 19).

The rod is used for breaking up any large pieces of cheese that may subsequently appear in the sample.

(16) "Official and Tentative Methods of Analysis of A.O.A.C.," 4th Edit., 1935, p. 290.

Add 1.5 ml. hot water and break up any lumps of cheese which do not disintegrate. Spread the cheese and water evenly over the entire bottom of the dish.

Place the dish in the "solids" hot plate operating at 180°C. and evaporate the contents to apparent dryness, using the dish depressor described in sec. A (1), p. 20.

Now transfer the dish to the "solids" vacuum oven operating at 105°C. and set the alarm clock for 20 minutes from the time the vacuum gauge shows a reading of 25".

After the expiry of this period transfer the dish to the "solids" desiccator again, setting the alarm clock for five minutes, taking especial care to see that the cooling fluid is flowing freely.

After the dish has cooled for the requisite period, transfer it to the balance and weigh rapidly.

Fat Determination.

Weigh accurately about 1.0 gram of the prepared sample for the estimation, employing either the butter boat method (sec. B (2*c*), p. 26) or the method of direct weighing into the fat extraction flask (sec. B (2*b*), p. 26).

First Extraction.

(1) Add 8 ml. of hot distilled water—cork and shake thoroughly.

Then (2) Add 3.0 ml. of ammonium hydroxide—cork and shake thoroughly.

,, (3) Add 10 ml. of alcohol—cork and shake for 20 seconds.

,, (4) Add 25 ml. of ethyl ether—cork and shake for 20 seconds.

,, (5) Add 25 ml. of petroleum ether—cork and shake for 20 seconds.

Keep the fat extraction flask securely corked and centrifuge for 30 seconds, the whole operation being completed in 30 turns of the centrifuge handle.

Uncork the flask carefully and decant the ethereal layer into the prepared "fats" dish and evaporate the ether (sec. B (3), p. 27, "First Extraction").

SECOND EXTRACTION.

(1) Add *no* distilled water.

(2) Add *no* ammonium hydroxide.

(3) Add 5 ml. alcohol—cork and shake for 20 seconds.

Then (4) Add 25 ml. ethyl ether—cork and shake for 20 seconds.

,, (5) Add 25 ml. petroleum ether—cork and shake for 20 seconds.

Keep the fat extraction flask securely corked and again centrifuge for 30 seconds, the whole operation being completed in 30 turns of the centrifuge handle.

Uncork the flask carefully and again decant the ethereal layer into the prepared "fats" dish and evaporate the ethers (sec. B (3), p. 27, "First Extraction").

If, as pointed out in sec. B (3), p. 27, "First Extraction," the line of separation of the liquids is too low, distilled water may be added just before the ethereal layer is poured off into the prepared "fats" dish during the second extraction.

After the evaporation of the ethers, heat the "fats" dish in the "fats" vacuum oven operating at 135°C., setting the alarm clock for five minutes from the time the gauge shows a reading of 25".

After the expiry of this period, remove the dish to the "fats" desiccator and again set the alarm clock for seven minutes, taking especial care to see that the cooling fluid is flowing freely.

When the dish has cooled for the requisite period transfer it to the balance and weigh rapidly.

MILK POWDERS (Includes Skim Milk, Buttermilk, Whole Milk and Whey Powders).

Method of Sampling.

(*a*) The sample, if not too large, may be divided into four equal parts after fairly thorough mixing. Individual samples are taken, one from each of these four quarters, and these subsidiary samples are well mixed together by placing them in a sample jar fitted with a well-fitting screw-cap lid and rotating the jar with occasional shaking.

The composite sample is then ready for analysis.

(*b*) For large consignments of Milk Powder the following method[17] can be utilized. On the surface of the milk powder select a point at each end of a diameter and on a radius perpendicular to the diameter 2″ from the edge of the barrel. Midway on each side of the triangle between these points, locate a point. At the six points so plotted, using a tubular trier sufficiently big to extend the full length of the barrel, draw a core parallel to the vertical axis of the barrel. The cores are combined, thoroughly mixed as rapidly as possible, and the composite sample is quartered as in (*a*) and the four samples so obtained are well mixed as rapidly as possible and placed in a screw-top sample jar. The whole of the above operations should be conducted as speedily as possible in order to avoid the powder absorbing moisture from the air.

Moisture Determination.

Weigh out accurately 2 grams of the prepared sample into the prepared moisture dish complete with lid (" Preparation of Dishes," p. 19), spreading the sample as evenly as possible over the entire surface of the dish.

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(17) " Official and Tentative Methods of Analysis of A.O.A.C.," 4th Edit., 1935, p. 282.

During the weighing operations the lid is placed on the balance pan with the dish resting on the top of it: when the amount required has been weighed out approximately the lid is quickly placed on the top of the dish and the exact final weight is then determined.

The whole is transferred by means of the special tongs directly to the " solids " vacuum oven operating at 105°C. and the alarm clock is set for one hour from the time the gauge records 25".

The lids must be kept in position throughout the heating period.

At the end of this period transfer the dish with the lid still in position quickly by means of the special tongs to the " solids " desiccator, setting the alarm clock for ten minutes: make sure that the cooling fluid is flowing freely.

When the dish has cooled for the requisite period transfer to the balance by means of the special tongs, with the lid still in position, and weigh as rapidly as possible.

Fat Determination.

Weigh accurately about 1 gram of the prepared sample into the fats extraction flask, employing either the butter boat method (sec. B (*2c*), p. 26) or the method of direct weighing (sec. B (*2b*), p. 26).

If preferred the sample can be weighed into a small beaker, dispersed in distilled water and the milk suspension so produced poured directly into the fat extraction flask, the beaker being washed out in turn with the reagents.

First Extraction.

(1) Add 8.5 ml. of hot distilled water—cork and shake thoroughly.

Then (2) Add 1.5 ml. ammonium hydroxide (3 ml. in case of buttermilk powder)—cork and shake thoroughly.

Then (3) Add 10 ml. alcohol—cork and shake for 30 seconds.
,, (4) Add 25 ml. ethyl ether—cork and shake for 20 seconds.
,, (5) Add 25 ml. petroleum ether—cork and shake for 20 seconds.

Keep the fat extraction flask securely corked and centrifuge for 30 seconds, the whole operation being completed in 30 turns of the centrifuge handle.

Uncork the flask carefully and decant the ethereal layer into the prepared "fats" dish and evaporate the ethers (sec. B (3), p. 27, "First Extraction").

SECOND EXTRACTION.

(1) Add *no* distilled water.
(2) Add *no* ammonium hydroxide.
(3) Add 5 ml. alcohol—cork and shake for 20 seconds.
Then (4) Add 15 ml. ethyl ether (25 ml. in the case of whole milk powder)—cork and shake for 20 seconds.
,, (5) Add 15 ml. petroleum ether (25 ml. in the case of whole milk powder)—cork and shake for 20 seconds.

Keep the fat extraction flask securely corked and again centrifuge for 30 seconds, the whole operation being completed in 30 turns of the centrifuge handle.

Uncork the flask carefully, again decant the ethereal layer into the prepared "fats" dish and evaporate the ethers (sec. B (3), p. 27, "First Extraction").

If, as pointed out in sec. B (3), p. 27, "First Extraction," the line of separation of the liquids is too low, distilled water may be added just before the ethereal layer is poured off into the prepared "fats" dish during the second extraction.

After the evaporation of the ethers heat the "fats" dish in the "fats" vacuum oven operating at 135°C., setting the alarm clock for five minutes from the time the gauge shows a reading of 25″.

After the expiry of this period, remove the dish to the "fats" desiccator and again set the alarm clock for seven minutes, taking especial care to see that the cooling fluid is flowing freely.

When the dish has cooled for the requisite period transfer it to the balance and weigh rapidly.

SPECIAL NOTE.—In the case of "spray" process milk powders it has been found necessary to make a third extraction: the technique employed for the second extraction should therefore be repeated.(18)

MALTED MILK POWDER, MILK and PLAIN CHOCOLATE, and COCOA.

Method of Sampling.

The method of sampling malted milk powders adopted is the same as for Milk Powders.

(*a*) The sample, if not too large, may be divided into four equal parts after fairly thorough mixing. Individual samples are taken one from each of these four quarters, and these subsidiary samples are well mixed together by placing them in a sample jar fitted with a well-fitting screw-cap lid, and rotating the jar with occasional shaking.

The composite sample is then ready for analysis.

(*b*) For large consignments of Malted Milk Powder the following method based upon that given in A.O.A.C. 4th Edition, 1935, p. 282, for milk powders can be utilized. On the surface of the powder select a point at each end of a diameter and on a radius perpendicular to the diameter 2″ from the edge of the barrel. Midway on each side of the triangle between these points, locate a point. At the six points so plotted, using a tubular trier

(18) Lampitt Hughes and Bogod, *Analyst*, 1924, **49**, 415.

sufficiently big to extend the full length of the barrel, draw a core parallel to the vertical axis of the barrel. The cores are combined, thoroughly mixed as rapidly as possible and the composite sample is quartered as in (*a*) and the four samples so obtained are well mixed as rapidly as possible and placed in a screw-top sample jar. The whole of the above operations should be conducted as speedily as possible in order to avoid the powder absorbing moisture from the air.

Sampling of Chocolate.

The block or tablet is sampled by removing thin slices with a small hand-plane, thoroughly mixing the shavings in a sample jar and breaking up the individual pieces as finely as possible without causing them to coalesce into larger pieces.

Sampling of Cocoa.

(*a*) If the cocoa is in the unfinished state, i.e., in the form of "cake" then the "cakes" are ground up in a pestle and mortar and thoroughly mixed.

The mixed powder is then quartered and portions removed from each quarter and thoroughly mixed. The resultant sample can be reduced in size if required by "quartering" again as just described, and removing portions as before.

(*b*) In the case of a prepared marketed cocoa the required sample is taken by the method of quartering just described.

Total Solids Determination.

Weigh accurately about 0.3 gram of the prepared sample directly into the prepared total-solids dish ("Preparation of the Dish," p. 19).

Add 2 ml. of hot distilled water to the sample in the dish and spread the mixture evenly over the entire bottom surface of the dish.

Place the dish and sample on the "solids" hot plate operating at 180°C. and evaporate the contents to apparent dryness using the dish depressor as described in sec. A (1), p. 20.

Now transfer the dish to the "solids" vacuum oven operating at 105°C. and set the alarm clock for 20 minutes from the time that the gauge registers 25″.

At the expiry of this period transfer the dish to the "solids" desiccator, again setting the alarm clock for five minutes, taking especial care to see that the cooling fluid is flowing freely.

After the dish has cooled for the requisite period transfer it to the balance and weigh rapidly.

Fat Determination.

Weigh accurately about 0.5 gram of the prepared sample into the fat extraction flask, employing either the butter boat method (sec. B (2*c*), p. 26), or the method of direct weighing (sec. B (2*b*), p. 26).

First Extraction.

(1) Add 8 ml. of hot distilled water—cork and shake thoroughly.

Then (2) Add 1.5 ml. ammonium hydroxide—cork and shake thoroughly.

,, (3) Add 10 ml. of alcohol—cork and shake for 30 seconds.

,, (4) Add 25 ml. of ethyl ether—cork and shake for 20 seconds.

,, (5) Add 25 ml. of petroleum ether—cork and shake for 20 seconds.

Keep the fat extraction flask securely corked and centrifuge for 30 seconds, the whole operation being completed in 30 turns of the centrifuge handle.

Uncork the flask carefully and decant the ethereal layer into the prepared "fats" dish and evaporate the ethers (sec. B (3), p. 27, "First Extraction").

SECOND EXTRACTION.

(1) Add *no* distilled water.
(2) Add *no* ammonium hydroxide.
(3) Add 5 ml. alcohol—cork and shake for 20 seconds.

Then (4) Add 25 ml. of ethyl ether—cork and shake for 20 seconds.

,, (5) Add 25 ml. of petroleum ether—cork and shake for 20 seconds.

Keep the fat extraction flask securely corked and again centrifuge for 30 seconds, the whole operation being completed in 30 turns of the centrifuge handle.

Uncork the flask carefully, again decant the ethereal layer into the prepared "fats" dish and evaporate the ethers (sec. B (3), p. 27, "First Extraction").

If, as pointed out in sec. B (3), p. 27, "First Extraction," the line of separation of the liquid is too low, distilled water may be added just before the ethereal layer is poured off into the prepared "fats" dish during the second extraction.

After the evaporation of the ethers heat the "fats" dish in the "fats" vacuum oven operating at 135°C., setting the alarm clock for five minutes from the time the gauge registers 25″.

After the expiry of this period, remove the dish to the "fats" desiccator and again set the alarm clock for seven minutes, taking especial care to see that the cooling fluid is flowing freely.

When the dish has cooled for the requisite period transfer it to the balance and weigh rapidly.

CHAPTER III.

SOME ADDITIONAL APPLICATIONS OF THE "TECHNICO" TEST UNIT TO THE DETERMINATION OF TOTAL SOLIDS.

Special Directions.

The thermostat governing the temperature of the hot plate must be so adjusted as to give a surface temperature of 100°C. for all total solids determinations other than those described in the foregoing pages.

All dishes used for total solids determinations are made of nickel, as aluminium dishes have been found under certain circumstances to corrode under the influence of the products evaporated in them.

Yeast and Cut Peels.

Method of Sampling.

(*a*) YEAST.—Take the usual 7 lb. bag and cut it into two equal portions across the short side. From the centre of each half, remove with a spatula the required amount of sample, mix thoroughly and place in a clean, dry, screw-top sample jar and keep the jar in a refrigerator until the sample is required for analysis.

(*b*) CUT PEELS.—The peel is removed from the four corners and the centre of the box, taking each sample completely to the bottom of the box. The samples so collected are intimately mixed and the composite sample obtained is again sampled to obtain the requisite amount for analysis.

This sample is then passed three times through a fine mincer, finally placed in a clean, dry, screw-top jar and kept in a cool place until required for analysis.

Preparation of the Dishes.

Proceed as described in Section "Preparation of the Dishes," p. 19.

Determination.

(*a*) YEAST.—Weigh directly into the prepared "solids" dish about 2 grams of the prepared sample, add about 5 ml. distilled water and work the yeast into a homogeneous cream with a flat-ended glass rod: wash off any adhering yeast with a small quantity of distilled water.

Place the dish carefully on the "solids" hot plate operating at 100°C. and carefully evaporate the contents to apparent dryness.

Transfer the dish to the "solids" vacuum oven operating at 105°C., and set the alarm clock for one hour from the time the gauge registers 25″.

After the expiry of this period, transfer the dish to the "solids" desiccator and again set the alarm clock for five minutes, taking especial care to see that the cooling fluid is flowing freely.

After the dish has cooled for the requisite time transfer to the balance and weigh rapidly.

(*b*) CUT PEELS.—Weigh directly into the prepared "solids" dish about 2 grams of the sample accurately: add about 5 ml. distilled water to the minced peel and spread evenly over the entire bottom surface of the dish.

Place the dish carefully on the "solids" hot plate operating at 100°C. and carefully evaporate the contents to apparent dryness.

Transfer the dish to the "solids" vacuum oven operating at 105°C. and set the alarm clock for one hour from the time the gauge indicates 25″.

Now transfer the dish and residue to the "solids" desiccator, and again set the alarm clock for five minutes, taking especial care to see that the cooling fluid is flowing freely.

After the dish has cooled for the requisite time, transfer to the balance and weigh rapidly.

Frozen Whole Eggs, Frozen Egg Yolks, Frozen Egg Albumin, Sugared Whole Egg and Sugared Yolks.

Method of Sampling.

(*a*) WHOLE FROZEN EGG.—The cans in which the eggs are usually packed are covered generally by a fabric-covered cardboard jacket: this jacket is removed and the cans to be sampled are placed, unopened in an upright position, in running water, preferably at about 70° F., for about 24 hours.

It is imperative that this "defrosting" operation should be carried out slowly.

At the end of this time the cans are opened and if any cores of frozen eggs remain, these are removed, defrosted separately, and returned to the appropriate cans. The contents of each can are then thoroughly mixed, preferably by hand, any lumps being carefully broken up and dispersed. Samples are taken from each can and these individual samples are again mixed thoroughly by means of a hand-plunger mixer before storing in sample screw-top jars.

The jars should then be placed in a refrigerator until the samples are required for analysis.

(*b*) FROZEN EGG YOLKS.—The cans in which the yolks are packed and which are usually covered as described under "Frozen Whole Eggs," are defrosted as described above in paragraph (*a*) for 24

hours. The defrosted yolks usually assume the texture of putty and thorough mixing is a difficult process: if mixing is impossible, samples should be taken from each can by means of a butter sampler, the cores being taken from the four corners and from the centre of each can. The cores are then passed through a mincer several times until a homogeneous sample is obtained. The composite samples so obtained are stored in screw-cap jars in a refrigerator until required for use.

(*c*) Frozen Egg Albumin.—The cans in which the albumin is packed are defrosted as described in paragraph (*a*).

The defrosted "whites" should be very thoroughly mixed by stirring, care being taken to avoid any tendency to beat or to incorporate air in any way.

The samples so obtained are stored in screw-cap jars and placed in a refrigerator until required for analysis.

(*d*) Sugared Whole Egg and Sugared Egg Yolks.—In the case of sugared whole egg and sugared yolks, these can be sampled easily by merely thoroughly mixing the contents of each can, and taking out the required portion and storing in a screw-top sample jar in a refrigerator until required for analysis.

Preparation of the Dishes.

Proceed as described in "Preparation of the Dishes," p. 19.

Determination.

(*a*) Frozen and Sugared Whole Egg.—Weigh directly and accurately into the prepared "solids" dish about 2 grams of the sample and spread it evenly over the bottom surface of the dish. Place the dish carefully on the "solids" hot plate operating at 100°C. and evaporate the contents to apparent dryness.

Transfer the dish to the " solids " vacuum oven operating at 105°C. and set the alarm clock for one hour from the time the gauge shows a reading of 25".

After the expiry of this period, transfer the dish to the " solids " desiccator, again setting the alarm clock for a period of ten minutes.

When the dish has cooled for the requisite period, transfer it to the balance and weigh rapidly.

(*b*) FROZEN EGG WHITES.—Weigh directly and accurately into the prepared " solids " dish about 2 grams of the sample and spread it evenly over the bottom surface of the dish.

Place the dish on the " solids " hot plate operating at 100°C. and evaporate the contents to apparent dryness.

Transfer the dish to the " solids " vacuum oven operating at 105°C. and set the alarm clock for one hour from the time the gauge shows a reading of 25".

After the expiry of this period transfer the dish to the " solids " desiccator and again set the alarm clock for a period of ten minutes.

When the dish has cooled for the requisite period, transfer it to the balance and weigh rapidly.

(*c*) FROZEN EGG YOLKS AND SUGARED EGG YOLKS.—Weigh directly and accurately into the prepared " solids " dish about 1 gram of the sample and spread it evenly over the bottom surface of the dish with about 5 ml. of distilled water.

Place the dish carefully on the " solids " hot plate operating at 100°C. and evaporate the contents to apparent dryness.

Transfer the dish to the " solids " vacuum oven operating at 105°C. and set the alarm clock for one hour from the time the gauge shows a reading of 25".

After the expiry of this period, transfer the dish to the " solids " desiccator, again setting the alarm clock for a period of ten minutes.

When the dish has cooled for the requisite period, transfer it to the balance and weigh rapidly.

Caramel ("Black Jack"), Glucose Syrup, Molasses, Golden Syrup, Black Treacle, Maple Syrup and Dried Fruit.

Method of Sampling.

(*a*) CARAMEL.—This product is usually homogeneous in character but nevertheless should be carefully mixed with a cream or milk plunger before the sample is taken; the sample should be stored in a screw-top sample jar.

(*b*) GLUCOSE.—No mixing should be necessary before taking the sample.

(*c*) MOLASSES.—No mixing should be necessary before taking the sample.

(*d*) GOLDEN SYRUP.—No mixing should be necessary before sampling.

(*e*) BLACK TREACLE.—No mixing should be necessary before sampling.

(*f*) MAPLE SYRUP.—If the sample contains no crystals then no mixing should be necessary before sampling.

If, on the other hand, the syrup contains crystalline or other solid matter transfer to a mortar and grind thoroughly until the whole is homogeneous.

Take the sample and store in a screw-top jar.

(*g*) DRIED FRUITS.—Remove any stones present (as in the case of prunes, for example) and pass the fruit through a mincer several times until homogeneous. Take a sample from the minced fruit and

store in a screw-top jar in a refrigerator until required for analysis: care must be taken that the sample does not freeze.

Preparation of the Dishes.

Proceed as described in "Preparation of the Dishes," p. 19.

Determination.

CARAMEL ("BLACK JACK"), GLUCOSE, MOLASSES, GOLDEN SYRUP, BLACK TREACLE, MAPLE SYRUP AND DRIED FRUITS.—Weigh directly and accurately into the prepared "solids" dish about 1-2 grams of the prepared sample: add about 5 ml. of water, rotate the dish gently until the liquid is homogeneous and spread the diluted sample evenly over the bottom surface of the dish.

In the case of dried fruits, mash up the sample in the 5 ml. of water with a flat-ended glass rod, taking care to wash off any adhering sample from the rod with the smallest amount of distilled water.

Place the dish carefully on the "solids" hot plate operating at 100°C. and evaporate the contents to apparent dryness.

Transfer the dish to the "solids" vacuum oven operating at 105°C. and set the alarm clock for a period of one hour from the time the gauge shows a reading of 25″.

After the expiry of this period, transfer the dish to the solids desiccator, taking especial care to see that the cooling fluid is flowing freely, and set the alarm clock again for a period of ten minutes.

When the dish has cooled for the requisite period transfer to the balance and weigh rapidly.

Fruit Products.

This includes Fresh and Frozen Fruits, Fruit in Syrup, Fruit Juices, Fruit Syrups, Canned Fruit, Fruit Juices ("Sulphited"), Fruit Pulps

(Canned), Fruit Pulps ("Sulphited"), Fruit Purée (Bottled), All types of Jams, Lemon Curd, Mincemeat.

Method of Sampling.

(*a*) FRESH FRUIT.—Firstly, stalks and stones should be carefully removed, and the remaining portion should be passed several times through a mincer until homogeneous: no losses of juice should be allowed. The minced sample is then thoroughly mixed, stored in a sample jar and kept in a refrigerator until required for analysis. On no account must the sample be allowed to freeze.

(*b*) FROZEN FRUIT.—All fruit preserved in this way should be defrosted very slowly, stalks and fruit stones should be removed, and the fruit should be passed, as described above, through a mincer, observing the precautions stated there.

(*c*) FRUIT IN SYRUP.—Firstly, all stalks, stones, leaves, etc., must be removed from the sample, after which the residue is passed as quickly as possible through the mincer, care being taken that none of the expressed juices are lost in the process.

(*d*) FRUIT JUICES (NOT "SULPHITED").—Stir the contents of the can or bottle thoroughly before sampling: no other form of mixing should be necessary. Store the sample in a refrigerator until required for analysis. On no account should the sample be allowed to freeze.

(*e*) FRUIT SYRUPS.—Sample and store in the same way as described under "(*d*) Fruit Juices." On no account must the sample be allowed to freeze.

(*f*) CANNED FRUIT IN WATER.—Such fruit as would fall under this heading would include "pack apples," blackcurrants in water, etc. No preparation, such as removal of cores, stalks, etc., is usually necessary.

An aliquot part of the contents of the can is taken and passed through the mincer several times until

the sample is homogeneous. Store in a screw-top jar in a refrigerator until required for analysis. On no account must the sample be allowed to freeze.

(*g*) FRUIT JUICES ("SULPHITED").—Mix the contents of the cask either by hand-plunging with a milk plunger or by rolling the cask repeatedly to and fro rapidly, so that no sulphur dioxide in any appreciable quantity is lost. Transfer the sample to a screw-top jar and store in a refrigerator until required for analysis. On no account must the sample be allowed to freeze.

(*h*) FRUIT PULP (CANNED).—Stir the contents of the can thoroughly, remove any stones, stalks, leaves, etc., take an aliquot portion, pass through a mincer several times until the sample is homogeneous, and store in a screw-top jar in a refrigerator until required for analysis. On no account must the sample be allowed to freeze.

(*i*) FRUIT PULP ("SULPHITED").—Such fruit as falls under this heading is usually stored in barrels. The "head" of the barrel should be removed, preferably by an experienced "cooper," and the contents should then be very thoroughly mixed by plunging with a milk plunger until the contents are completely mixed. The sample should be taken quickly while the pulp is still agitated so that it is representative of the whole.

Stones, stalks, etc., should be removed from the sample so taken and then it is preferable to separate roughly the "liquor" from the solid fruit by means of a 40-mesh sieve. The solid fruit is next minced several times until homogeneous, taking care that no expressed "liquor" is lost: finally, combine the minced and "liquor" fractions, mix thoroughly, store in a screw-top jar and keep in a refrigerator until required for analysis. On no account must the sample be allowed to freeze.

(*j*) FRUIT PURÉE (BOTTLED).—The contents of the bottle must be thoroughly shaken to ensure even

distribution of any particles of sand, the presence of which is not infrequent, before a sample is taken.

Remove an aliquot portion of the contents, store in a screw-top jar and keep in a refrigerator until required for analysis. On no account must the contents be allowed to freeze.

(*k*) JAMS.—Any stones, leaves, etc., should be removed. If the jam is a whole-fruit product the contents of the container should be passed through a mincer until absolutely homogeneous, after which a sample is taken and stored in a screw-cap jar in a refrigerator until required for analysis.

If the jam is not whole-fruit in nature thorough mixing with a strong wooden spatula should prove adequate.

(*l*) LEMON CURD.—The contents of the container should be thoroughly stirred before sampling. The sample should be stored in a screw-cap jar and kept in a refrigerator until required for analysis. The sample must on no account be allowed to freeze.

(*m*) MINCEMEAT.—Very thorough stirring of the contents of the container must be carried out to ensure that any liquid fraction which may have separated is dispersed evenly throughout the mass. The sample is taken from the mass and passed through a mincer several times before any analysis is undertaken: it is then stored in a screw-cap jar and kept in a refrigerator until required for analysis. The sample must on no account be allowed to freeze.

Preparation of the Dishes.

Proceed as described in Section "Preparation of the Dishes," p. 19.

Determination.

The method adopted by Hughes and Maunsell has been found successful.[19]

(19) *Analyst*, 1934, **59**, 231.

As pointed out in this paper, errors are introduced by the inversion of cane sugar during drying, if present in appreciable amounts, due to the presence of the fruit acids. Hughes and Maunsell state that the trouble can be overcome by the neutralisation of the acids before evaporation of the sample to dryness.

It therefore becomes necessary to determine the soluble solids concentration of the sample before carrying out the total solids determination, and this can be done quickly and reasonably accurately by the use of the refractometer.

If the soluble solids content of the sample exceeds the figure of 15%, then neutralisation becomes necessary, but soluble solids figures falling much below 15% render neutralisation unnecessary.

(*Soluble Solids Figure below 15%*).

Weigh accurately about 2 grams of the sample into the prepared " solids " dish, add about 5 ml. of distilled water and spread evenly over the bottom of the dish.

Place the dish on the " solids " hot plate operating at 100°C. and evaporate to apparent dryness.

Transfer the dish to the " solids " vacuum oven operating at 105°C. and set the alarm clock for 30 minutes from the time the gauge shows a reading of 25".

Now cool the dish in the " solids " desiccator, again setting the alarm clock for five minutes. Take especial care to see that the cooling fluid is flowing freely. After the dish has cooled for the requisite period transfer to the balance and weigh rapidly.

(*Solids Figure above 15%*).

Neutralisation (based on Method of Hughes and Maunsell).[19]

Weigh accurately about 10 grams of the sample into a litre capacity conical flask, add about 200-300

(19) *Loc. cit.*

ml. distilled water and boil gently for about a quarter of an hour, adding distilled water from time to time to keep the volume reasonably constant.

Cool the contents to room temperature and titrate with 0.5*N* standard sodium hydroxide, using phenol phthalein as an internal indicator. About 0.5 ml. of indicator should be used.

Now weigh accurately about 2 grams of sample into the prepared "solids" dish ("Preparation of the Dishes," p. 19), and add from a burette the amount of 0.1*N* sodium hydroxide required for the weight of sample taken, calculated from the titration figure obtained above.

Spread the sample evenly over the bottom of the dish, mixing thoroughly with a glass rod to ensure complete neutralisation of the fruit acids by the added sodium hydroxide. Carefully wash off any sample adhering to the glass rod with distilled water.

Place the dish on the "solids" hot plate operating at 100°C. and evaporate to apparent dryness.

Transfer the dish to the "solids" vacuum oven operating at 105°C. and set the alarm clock for a period of 30 minutes from the time the gauge registers 25″.

After the expiry of this period, transfer the dish to the solids desiccator and again set the alarm clock for a period of five minutes: make sure that the cooling fluid is flowing freely.

When the dish has cooled for the requisite period transfer to the balance and weigh rapidly.

Correction to be applied.

A control determination is made in the following manner. Take 2 grams A.R. citric acid accurately weighed and make up to 200 ml. Pipette out 20 ml. of this solution and neutralise to 0.1*N* sodium hydroxide, using one or two drops of phenol

phthalein as an internal indicator, boiling the solution in the same way and for the same time as the sample was boiled.

Pipette out into a prepared "solids" dish ("Preparation of the Dishes," p. 19), 5 ml. of the 1% citric acid solution and add carefully one quarter the amount of 0.1*N* sodium hydroxide required in the original titration.

Transfer the dish to the "solids" hot plate operating at 100°C. and evaporate to apparent dryness.

Transfer the dish to the "solids" vacuum oven operating at 105°C. and set the alarm clock for 30 minutes from the time the gauge registers 25″.

After the expiry of the period, cool the dish in the "solids" desiccator for five minutes as determined by the alarm clock, making sure that the cooling fluid is flowing freely.

When the dish has cooled for the requisite period, transfer to the balance and weigh rapidly.

The weight of citric acid contained in 5 ml. of solution, i.e., 0.05 gram, subtracted from the total weight of residue gives the increase in weight due to neutralisation and hence the increase in weight per ml. of standard alkali can be calculated.

The increase, therefore, due to the amount of alkali added in the determination is subtracted from the determined total solids weight and the percentage total solids in the sample can then be calculated.

French and German Mustards and Vinegars (All Types).

Method of Sampling.

(*a*) French and German Mustards. — A thorough mixing with a wooden or nickel spatula should prove adequate. The sample should be kept

in a small screw-cap jar and stored in a refrigerator until required for analysis. The sample should not be allowed to freeze.

(*b*) VINEGARS.—The bottle should be well shaken or the cask should be rolled thoroughly before a sample is taken.

Store in a screw-cap jar and keep in a refrigerator until required for analysis. The sample must not be allowed to freeze.

Preparation of the Dishes.

Proceed as described under "Preparation of the Dishes," p. 19.

Determination.

(*a*) FRENCH AND GERMAN MUSTARDS.—Weigh accurately about 2 grams of the sample into the prepared dish, add about 5 ml. of distilled water and spread the sample evenly over the bottom of the "solids" dish, ensuring thorough mixing by using a flat-ended glass rod. Wash off any of the sample adhering to the glass rod into the dish by means of distilled water.

Place the dish on the "solids" hot plate operating at 100°C. and evaporate to apparent dryness.

Transfer the dish to the "solids" vacuum oven operating at 105°C., and set the alarm clock for 40 minutes from the time the gauge registers 25″.

After the expiry of this period cool the dish in the "solids" desiccator, setting the alarm clock for a period of five minutes: make sure that the cooling fluid is flowing freely.

After the dish has cooled for the requisite period, transfer the dish to the balance and weigh rapidly.

(*b*) VINEGARS (INCLUDES MALT, WINE, DISTILLED AND SPIRIT VINEGARS).—Weigh accurately and rapidly about 10-15 grams of the sample into the prepared dish and spread it evenly over the bottom surface of the dish.

Place the dish on the " solids " hot plate operating at 100°C. and evaporate to apparent dryness.

Transfer the dish to the " solids " vacuum oven operating at 105°C. and set the alarm clock for 40 minutes from the time the gauge registers 25".

Cool the dish in the " solids " desiccator, setting the alarm clock for ten minutes and making sure that the cooling fluid is flowing freely.

After the dish has cooled for the requisite period, transfer to the balance and weigh rapidly.

Sauces, Pickles, Chutney and Tomato Purée.

Method of Sampling.

(*a*) SAUCES.—In most cases, sauces can be satisfactorily sampled by vigorous shaking of the containers or by emptying the contents into a cylindrical milk-powder reconstitution apparatus and plunging well with the plunger: a portion of the well-mixed material is placed in a screw-cap jar and stored in a refrigerator until required for analysis. Care must be taken to avoid freezing of the sample.

(*b*) PICKLES AND CHUTNEY.—If the product contains pieces of solid matter, the whole of the contents of the container is passed through a mincer several times until a homogeneous product is obtained. A portion is taken of the well-mixed sample so prepared and stored in a screw-cap jar in a refrigerator until required for analysis. On no account must the sample be allowed to freeze.

(*c*) TOMATO PURÉE.—The contents of the container are tipped out completely into a bowl and are thoroughly mixed by stirring. A sample is taken from this well mixed product and stored in a screw-cap jar in a refrigerator. Care must be taken that the sample does not freeze.

Preparation of the Dishes.

Proceed as described under section "Preparation of the Dishes," p. 19.

Determination.

Weigh accurately about 2-3 grams of the sample into the prepared dish and add about 5 ml. of distilled water: spread the sample evenly over the bottom of the dish, if necessary with a glass rod, taking care to wash off any material adhering to the glass rod with a small amount of distilled water.

Place the dish on the "solids" hot plate operating at 100°C. and evaporate the contents to apparent dryness.

Now transfer the dish to the "solids" vacuum oven operating at 105°C., setting the alarm clock for 35 minutes from the time the gauge registers 25".

Cool the dish in the "solids" vacuum desiccator, setting the alarm clock for ten minutes, taking care that the cooling fluid is flowing freely.

After the dish has cooled adequately, remove to the balance and weigh rapidly.

Honey.

Method of Sampling.

The sample should be stirred thoroughly, but not too vigorously so that air is not included. If the product is crystalline in nature the container should be placed in warm water at about 30°C. and the contents stirred until the crystals have disappeared: the product should then be cooled and a sample taken and kept in a screw-cap jar until required for analysis.

Preparation of the Dishes.

Proceed as described under section "Preparation of the Dishes," p. 19.

Determination.

Weigh accurately 2 grams of the sample into the prepared dish and add 5 ml. hot distilled water: mix thoroughly with a glass rod and spread the sample evenly over the bottom of the dish. Wash off any adhering sample from the rod with the minimum amount of hot distilled water and place the dish on the " solids " hot plate operating at 100°C.: evaporate the contents to apparent dryness.

Transfer the dish to the " solids " vacuum oven operating at 105°C. and set the alarm clock for one hour from the time the gauge records a reading of 25".

Cool the dish in the " solids " desiccator, setting the alarm clock for ten minutes, taking care that the cooling fluid is flowing freely.

After the dish has cooled for the requisite period remove to the balance and weigh rapidly.

Candied Peels and Glacé Fruits.

Method of Sampling.

The products falling under this head should be passed through a mincer several times until absolutely homogeneous.

A sample is taken from the minced product and stored in a screw-cap jar until required for analysis.

Preparation of the Dishes.

Proceed as described under section " Preparation of the Dishes," p. 19.

Determination.

Weigh out 1-2 grams of the sample accurately into the prepared " solids " dish and add 5 ml. hot distilled water. Mix the sample and the water thoroughly together and spread evenly over the bottom surface of the dish.

Place the dish on the " solids " hot plate operating at 100°C. and evaporate the contents to apparent dryness.

Transfer the dish to the "solids " vacuum oven operating at 105°C. and set the alarm clock for 45 minutes from the time the gauge records a reading of 25".

Cool the dish in the " solids " desiccator, setting the alarm clock for a period of ten minutes and taking care that the cooling fluid is flowing freely.

After the dish has cooled for the requisite period, remove to the balance and weigh rapidly.

Fondant and Toffee.

Method of Sampling.

(*a*) FONDANT.—The fondant under examination should be softened by warming in a container partly immersed in a water bath at 98-100°F. (34-38°C.) and stirring well until the whole is thoroughly mixed. Take a portion and store in a screw-cap jar and cool in a room at a moderate temperature for at least 12 hours.

(*b*) TOFFEE.—Several individual pieces should be selected and the outside portion should be cut off by means of a sharp penknife or scalpel.

The inside portions only should be used for analysis.

Preparation of the Dishes.

Proceed as described under section " Preparation of the Dishes," p. 19.

Determination.

Weigh accurately ten grams of the sample, dissolve in distilled water and make up the volume to 200 ml. in a graduated flask. Pipette out 10 ml.

of this dilute solution into the prepared dish and place the dish on the " solids " hot plate operating at 100°C. and evaporate the contents to apparent dryness.

Transfer the dish to the " solids " vacuum oven operating at 105°C., setting the alarm clock for 90 minutes from the time the gauge shows a reading of 25".

Cool the dish in the " solids " desiccator, setting the alarm clock for ten minutes: make sure that the cooling fluid is flowing freely.

After the dish has cooled for the requisite period transfer to the balance and weigh rapidly.

Turkish Delight and Marshmallow.

Method of Sampling.

(*a*) TURKISH DELIGHT.—Pass the contents of the container through a mincer several times until homogeneous: take the sample from the minced product and store in a screw-cap jar until required for analysis.

(*b*) MARSHMALLOW.—Sample and store in the same manner as described for Turkish Delight.

Preparation of the Dishes.

Proceed as described under section " Preparation of the Dishes," p. 19.

Determination.

Weigh accurately about 1-1.5 grams of sample into the prepared " solids " dish, add about 5 ml. of distilled water and mash up with a flat-ended glass rod, spreading the sample evenly over the bottom of the solids dish: care must be exercised that all sample adhering to the glass rod is washed back into the " solids " dish by means of a small amount of distilled water.

Place the dish on the " solids " hot plate operating at 100°C. and evaporate the contents to apparent dryness.

Transfer the dish to the "solids " vacuum oven operating at 105°C. and set the alarm clock for 90 minutes from the time the gauge records a reading of 25".

Cool the dish in the " solids " desiccator, setting the alarm clock for ten minutes, and taking especial care that the cooling fluid is flowing freely.

After the dish has cooled for the requisite period, transfer to the balance and weigh rapidly.

Pound & Slab Fruit Cakes (Genoa, Cherry, etc.), Swiss Rolls, Jam and Cream-Filled Sponge Goods, etc.

Method of Sampling.

(*a*) Pound Fruit Cakes.—The cake selected should be passed completely through a mincer several times until the resultant product is absolutely homogeneous. A portion is then chosen and stored in a screw-cap jar in a refrigerator until required for analysis. The jar must be completely filled with the sample.

(*b*) Slab Fruit Cakes.—The slab selected is cut in such a way that the sample taken contains a similar proportion of crust and crumb as that existing in the original cake. This portion is then minced several times until absolutely homogeneous. This is then stored in a screw-cap jar in a refrigerator until required for analysis. The jar must be completely filled with the sample.

(*c*) Swiss Rolls, Jam and Cream-Filled Sponge Goods, etc.—The complete article is passed through a mincer several times until an

absolutely homogeneous mass is obtained. The product is stored in a screw-cap jar in a refrigerator until required for analysis. The jar must be completely filled with the sample.

Preparation of the Dishes.

Proceed as described under section "Preparation of the Dishes," p. 19.

Determination.

Weigh accurately about 2 grams of the sample into the prepared "solids" dish and add about 5 ml. of distilled water. Mix the sample and the water thoroughly with a glass rod, spreading it evenly over the bottom surface of the dish and wash off any sample adhering to the glass rod by means of the smallest possible quantity of distilled water.

Place the dish on the "solids" hot plate operating at 100°C. and evaporate the contents to apparent dryness.

Transfer the dish to the "solids" vacuum oven operating at 105°C. and set the alarm clock for one hour from the time the gauge registers a reading of 25″.

Cool the dish in the "solids" desiccator, setting the alarm clock for ten minutes and making sure that the cooling fluid is flowing freely.

After the dish has cooled for the requisite period transfer it to the balance and weigh rapidly.

CHAPTER IV.

SOME APPLICATIONS OF THE "TECHNICO" TEST UNIT TO THE DETERMINATION OF MOISTURE.

In all the determinations which follow the dishes, which should be of aluminium, containing the weighed sample, are kept covered by their appropriate lids, also made of aluminium, from the time they enter the "solids" vacuum oven until they are finally weighed. This precaution is extremely important and is a step in the method which should on no account be omitted (see p. 19).

Flour, Starches, Salt.

Method of Sampling.

(*a*) FLOUR.—The method based on that advocated in "Cereal Laboratory Methods"[20] 3rd Edition, p. 28, is considered most suitable.

The number of bags selected as suitable for sampling should comprise the square root of the number making up the delivery.

The actual bags selected for sampling should consist of four taken from the most exposed, three from the next less exposed, two from the next, two from the next and one from the least exposed, from every lot selected, or in this ratio for smaller lots.

Into each bag to be sampled insert a pointed polished metal trier, $\frac{1}{2}''$ in diameter, with a slit at least one-third the circumference, from one corner

(20) "Official and Tentative Methods of Analysis of A.O.A.C.," 3rd Edit., 1930, p. 28. Also *J. Amer. Assoc. Off. Agric. Chem.*, 1925, 8, 424. *J. Amer. Assoc. Off. Agric. Chem.*, 1926, 9, 423.

of the top diagonally to the centre. Draw a second core from the other top corner to half the distance to the centre of the bag.

Deliver the two cores at once to a clean, dry, air-tight container which has stood open for a few minutes near the lot of flour to be sampled and seal immediately. Use a separate container for each bag sampled.

If the sample is too large the size should be reduced by "quartering." Spread the sample and divide into four quarters, discard the opposite quarters and mix again. Repeat until the sample is of the required size.

When it is necessary to store samples previous to analysis the container used should be of such a size that the sample completely fills it. The sample should be thoroughly mixed with a spatula just previous to weighing for analysis.

(*b*) STARCHES (i.e. CORNFLOUR, ARROWROOT, ETC.).—If the starch is delivered in sacks, sampling can be carried out in the same way as outlined in the case of flour.

If the starch is delivered in a keg, or circular tin, samples should be taken by means of the flour sampler from each of the four points lying on the diameters drawn at right-angles to each other and distant about two inches from the circumference.

If the container is square, samples should be taken by means of the flour sampler from each of the four corners.

The cores should be taken right through the mass of the product and the starch so withdrawn bulked together quickly on Kraft paper: the mixing should be carried out by continuously rolling the paper from each corner to the centre in turn until complete mixing has been accomplished.

If the resultant sample is too large it is reduced by "quartering" as described under flour, until the final sample is of the desired proportions.

As in the case of flour the sample jar should be completely filled and the screw-cap securely fastened on.

(*c*) COMMON SALT.—This product is usually delivered in sacks and should wherever possible be sampled as described in the case of flour.

The sample jar should be completely filled and the screw-cap securely replaced.

All sampling operations should be conducted with the greatest speed possible on account of the somewhat hygroscopic nature of the product under examination.

Preparation of the Dishes.

Proceed as described in section "Preparation of the Dishes," p. 19.

Determination.

Weigh accurately about 2 grams of the substance under examination into the prepared dish complete with lid: during the weighing operations the lid is placed on the balance pan with the dish resting on the top of it: when the amount required has been weighed out approximately the lid is quickly placed on the top of the dish and the exact final weighing is then completed. The whole is then transferred by means of the special tongs directly to the "solids" vacuum oven operating at 105°C., and the alarm clock is set for one hour from the time the gauge records 25″.

The lids should be kept in position throughout the heating period.

At the end of this period transfer the dish, *with the lid still in position,* quickly by means of the special tongs to the "solids" desiccator, setting the alarm clock for ten minutes make sure that the cooling fluid is flowing freely.

When the dish has cooled for the requisite period transfer to the balance by means of the special tongs, *with the lid still in position,* and weigh as rapidly as possible.

Bread and Sponge Cakes.

Method of Sampling.

(*a*) BREAD.—Select a loaf which is representative of the batch whence it is taken and cut the loaf in such a manner that the selected sample contains a similar proportion of crumb and crust as that existing in the original loaf.

The selected portion is cut into slices about $\frac{1}{8}$" thick, placed well spaced out on a weighed metal tray and the weight obtained. This tray is then exposed to ordinary room temperature for about 24 hours, after which the tray and air-dried bread are again weighed.

This loss in weight from a known weight of bread is recorded.

The air-dried slices are then ground gently in a mortar to a fairly fine powder, care being taken that heat is not generated which would introduce errors.

The ground sample is stored in a screw-cap jar, which is completely filled with the ground sample.

(*b*) SPONGE CAKES.—Select representative cakes from the batch or from the carton and slice the cakes thinly with a sharp knife.

These slices are then air-dried as described in the case of bread and the loss in weight for a known weight of sponge is obtained.

Preparation of the Dishes.

Proceed as described under section "Preparation of the Dishes," p. 19.

Determination.

Weigh accurately about 2 grams of the ground, air-dried sample in the prepared dish and lid: during the weighing operations the lid should be placed on the balance pan with the dish resting on the top of it: when the sample has been weighed out approximately the lid is quickly placed on the top

of the dish and the exact final weighing is completed. The whole is then transferred by means of the special tongs directly to the "solids" vacuum oven operating at 105°C., and the alarm clock is set for one hour from the time the gauge records 25". *The lid should be kept in position throughout the heating period.*

At the end of the period transfer the dish, *with the lid still in position,* quickly by means of the special tongs to the "solids" desiccator, setting the alarm clock for seven minutes and making sure that the cooling fluid is flowing freely.

When the dish has cooled for the requisite period transfer to the balance case by means of the special tongs, *with the lid still in position,* and weigh as rapidly as possible.

The loss in weight during this last drying together with the loss in weight during air-drying gives the total moisture loss.

Plain Cake (Madeira, Pound and Slab Cakes).

Method of Sampling.

(*a*) Pound Cakes.—In the case of pound cakes, these should be completely crumbed by hand as finely as possible: the crumbed cake should then be thoroughly mixed on a piece of Kraft paper by continuously rolling the paper from each corner to the centre in turn until complete mixing has been accomplished.

If the resulting sample is too large it is reduced by "quartering," as described under flour, until the final sample is of the desired proportions.

The sample jar in which the sample is stored should be completely filled and should be kept in a refrigerator until required for analysis.

(*b*) Slab Cake.—A complete slab should be selected and cut in such a way that as in the case

of bread, the selected sample contains a similar proportion of crumb and crust as that existing in the original cake.

This portion is crumbed by hand, taking care that too much pressure between the fingers does not cause the crumb to "ball" together.

The crumbed sample is then thoroughly mixed as quickly as possible by the method described above and if necessary the final sample obtained by "quartering" in the manner given above.

The chosen sample is stored in screw-cap jars, completely filled, in a refrigerator until required for analysis.

Preparation of the Dishes.

Proceed as described under section "Preparation of the Dishes," p. 19.

Determination.

Weigh accurately about 2 grams of the sample into the prepared dish and lid.

During the weighing operations the lid should be placed on the balance pan with the dish resting on the top of it: when the sample has been weighed out approximately the lid is quickly placed on the top of the dish and the exact final weighing is completed. The whole is then transferred, by means of the special tongs, directly to the "solids" vacuum oven, operating at 105°C., setting the alarm clock for one hour from the time the gauge registers a reading of 25". *The lid should be kept in position throughout the heating period.*

At the end of this period transfer the dish, *with the lid still in position,* quickly by means of the special tongs to the "solids" desiccator, setting the alarm clock for seven minutes and making sure that the cooling fluid is flowing freely.

When the dish has cooled for the requisite period transfer it by means of the special tongs, *with the lid still in position,* to the balance and weigh as rapidly as possible.

Biscuits, Baked Puff and Short Pastry.

Method of Sampling.

A representative number of biscuits or articles of puff and short pastry are taken and powdered in a mortar. Care must be exercised in this operation that undue pressure is not exerted by the pestle as excessive pressure or prolonged grinding causes separation of fat, especially in the cases of shortbread and butter puff biscuits and biscuits of a similarly high fat content.

The powdered sample is stored in a screw-cap jar and kept in a refrigerator until required for analysis. The sample jar must be completely filled.

Preparation of the Dishes.

Proceed as described under section "Preparation of the Dishes," p. 19.

Determination.

Weigh accurately about 2 grams of the powdered sample into the prepared moisture dish and lid. During the weighing operations the lid should be placed on the balance pan with the dish resting on the top of it: when the sample has been weighed out approximately, the lid is placed quickly on the top of the dish and the exact final weighing is completed. The whole is then transferred by means of the special tongs directly to the "solids" vacuum oven operating at 105°C., setting the alarm clock for one hour from the time the gauge records a reading of 25". *The lid should be kept in position throughout the heating period.*

At the end of this period transfer the dish, *with the lid still in position,* by means of the special tongs, to the " solids " desiccator, quickly setting the alarm clock for ten minutes and making sure that the cooling fluid is flowing freely.

When the dish has cooled for the requisite period transfer it, *with the lid still in position,* by means of the special tongs to the balance and weigh as rapidly as possible.

APPENDIX.

Tabulated Data for Use with the Technico Test Unit.

FAT DETERMINATIONS.

Substance.	Weight taken. grams.	Time in Vacuum Oven. mins.	Time in Desiccator. mins.	Remarks.	Reference. Page.
Fresh Milk . .	10	5	7	—	35
Skim Milk . .	10	5	7	—	35
Whey . . .	10	5	7	—	35
Buttermilk . .	10	5	7	—	35
Cream (less than 25% fat) . .	2	5	7	Add 5 ml. distilled water.	38
Cream (greater than 25% fat)	1	5	7	Add 6 ml. distilled water.	38
Unsweetened Cond. Milk .	5	5	7	—	42
Evaporated Milk	5	5	7	—	42
Condensed Buttermilk .	3	5	7	—	42
Sweetened Condensed Milk .	5	5	7	—	45
Ice Cream Mix .	5	5	7	—	48
Butter . . .	1	5	7	—	51
Cheese . . .	1	5	7	—	54
Skim Milk Powder	1	5	7	—	57
Buttermilk Powder	1	5	7	—	57
Whole Milk Powder . .	1	5	7	—	57
Whey Powder .	1	5	7	—	57
Malted Milk Powder . .	0.5	5	7	—	61
Milk Chocolate .	0.5	5	7	—	61
Plain Chocolate .	0.5	5	7	—	61
Cocoa . . .	0.5	5	7	—	61

TOTAL SOLIDS.

Substance.	Weight taken. grams.	Time in Vacuum Oven. mins.	Time in Desiccator. mins.	Remarks.	Reference. Page.
Fresh Milk . .	2	10	5	Add no water.	34
Skim Milk . .	2	10	5	,,	34
Whey	2	10	5	,,	34
Buttermilk . .	2	10	5	,,	34
Cream (less than 25% fat) . .	1	10	5	Add 1 ml. water.	37
Cream (more than 25% fat) . .	0.5	10	5	Add 1.5 ml. water.	37
Unsweetened Cond. Milk .	1	10	5	Add 1 ml. water.	41
Evaporated Milk	1	10	5	,,	41
Condensed Buttermilk .	0.5	10	5	Add 2 ml. water.	41
Sweetened Condensed Milk* .	0.25	90	5	Add 2 ml. hot water.	44
Ice Cream Mix .	1	10	5	Add 1 ml. water.	48
Butter	1	10	5	Add no water.	51
Cheese . . .	0.5	20	5	Add 1.5 ml. hot water.	53
Malted Milk Powder . .	0.3	20	5	Add 2 ml. hot water.	60
Plain Chocolate .	0.3	20	5	,,	60
Milk Chocolate .	0.3	20	5	,,	60

* Note.—An alternative and more rapid method is also given on page 45.

TOTAL SOLIDS—(*Continued*).

Substance.	Weight taken. grams.	Time in Vacuum Oven. mins.	Time in Desiccator. mins.	Remarks.	Reference. Page.
Cocoa Powder .	0.3	20	5	Add 2 ml. hot water.	60
Yeast (dried) .	2	60	5	Add 5 ml. water.	64
Cut Peels . .	2	60	5	,,	64
Frozen Whole Eggs . . .	2	60	10	Add no water.	66
Frozen Egg Yolks . . .	1	60	10	Add 5 ml. water.	67
Frozen Egg Whites . . .	2	60	10	Add no water.	67
Sugared Whole Egg . . .	2	60	10	,,	66
Sugared Yolks .	1	60	10	Add 5 ml. water.	67
Caramel . . .	1-2	60	10	,,	69
Glucose Syrup .	1-2	60	10	,,	69
Molasses . . .	1-2	60	10	,,	69
Golden Syrup .	1-2	60	10	,,	69
Black Treacle .	1-2	60	10	,,	69
Maple Syrup .	1-2	60	10	,,	69
Dried Fruit . .	1-2	60	10	,,	69
Fresh and Frozen Fruits . . .	2	30	5	See special remarks re neutralisation.	72 to 75
Fruit in Syrup .	2	30	5	,,	72-75
Fruit Juices . .	2	30	5	,,	72-75
Fruit Syrups . .	2	30	5	,,	72-75
Canned Fruit .	2	30	5	,,	72-75

TOTAL SOLIDS—(*Continued*).

Substance.	Weight taken. grams.	Time in Vacuum Oven. mins.	Time in Desiccator. mins.	Remarks.	Reference. Page.
Fruit Juices (Sulphited) .	2	30	5	See special remarks re neutralisation.	72 to 75
Fruit Pulps (Canned) . .	2	30	5	,,	72-75
Fruit Pulps (Sulphited) .	2	30	5	,,	72-75
Fruit Purée (Bottled) . .	2	30	5	,,	72-75
Jams	2	30	5	,,	72-75
Lemon Curd . .	2	30	5	,,	72-75
Mincemeat . .	2	30	5	,,	72-75
French Mustard .	2	40	5	Add 5 ml. distilled water.	76
German Mustard	2	40	5	,,	76
Malt Vinegar .	10-15	40	10	—	76
Wine Vinegar .	10-15	40	10	—	76
Distilled Vinegar	10-15	40	10	—	76
Spirit Vinegar .	10-15	40	10	—	76
Sauces . . .	2-3	35	10	Add 5 ml. distilled water.	78
Pickles . . .	2-3	35	10	,,	78
Chutney . . .	2-3	35	10	,,	78
Tomato Purée .	2-3	35	10	,,	78
Honey . . .	2	60	10	,,	79
Candied Peels .	1-2	45	10	,,	79
Glacé Fruits . .	1-2	45	10	,,	79
Fondant . . .	10 ml. of 5% solution	90	10	See special remarks on.	80 81

TOTAL SOLIDS—(*Continued*).

Substance.	Weight taken. grams.	Time in Vacuum Oven. mins.	Time in Desiccator. mins.	Remarks.	Reference. Page.
Toffee . . .	10 ml. of 5% solution	90	10	See special remarks on	80 to 81
Turkish Delight .	1-1.5	90	10	Add 5 ml. distilled water.	81
Marshmallow .	1-1.5	90	10	,,	81
Pound and Slab Fruit Cakes .	2	60	10	,,	83
Swiss Rolls . .	2	60	10	,,	83
Jam and Cream-Filled Sponge Goods . . .	2	60	10	,,	83

MOISTURES.

Substance.	Weight taken. grams.	Time in Vacuum Oven. mins.	Time in Desiccator. mins.	Reference. Page.
Flour	2	60	10	87
Starches	2	60	10	87
Salt	2	60	10	87
Bread	2*	60	7	88
Sponge Cakes . .	2*	60	7	88
Plain Cake (Madeira, etc.).	2	60	7	90
Biscuits	2	60	10	91
Baked Puff Pastry .	2	60	10	91
Baked Short Pastry	2	60	10	91
Skim Milk Powder .	2	60	10	56
Buttermilk Powder	2	60	10	56
Wholemilk Powder	2	60	10	56
Whey Powder . .	2	60	10	56

* The determination is carried out on the air-dried sample.

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